

OCTOBER, 1958

No. 213



# Bulletin

**Preview of ASTM Publications for 1958-1959**

**American Society for Testing Materials**



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# ASTM BULLETIN

October 1958

Number 233

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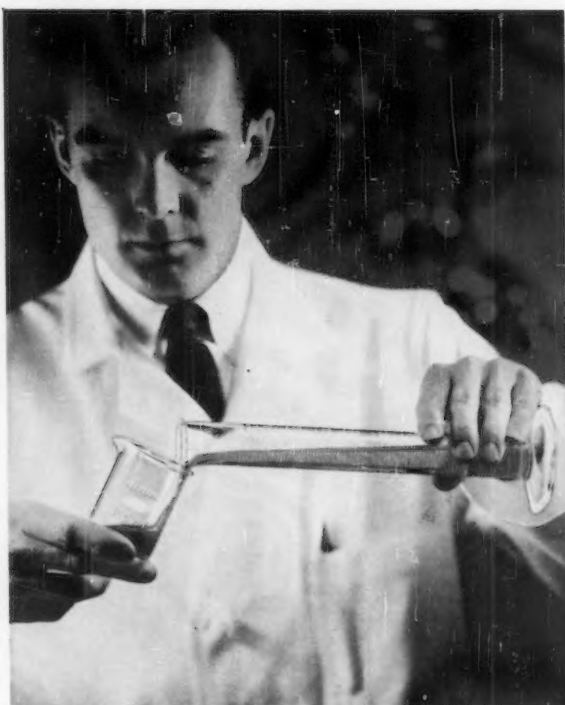
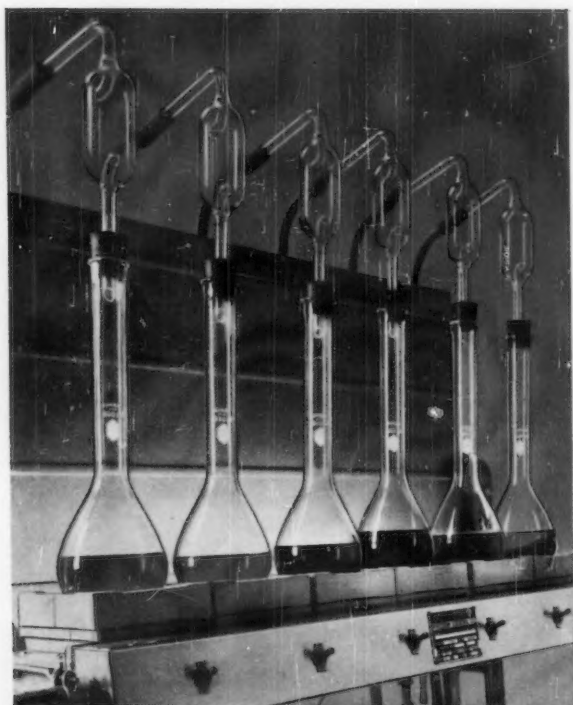
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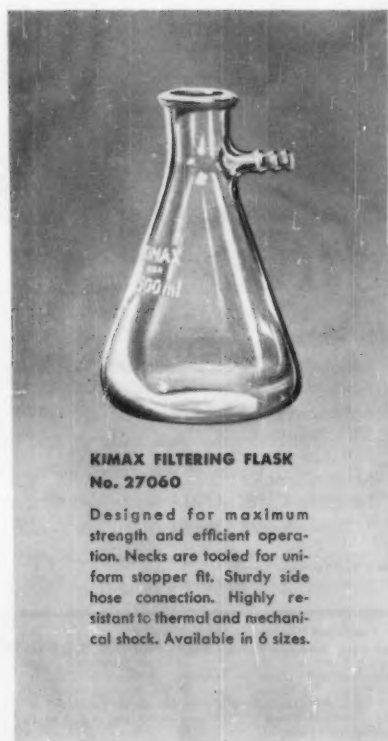
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## From corrosive alkali to biting acid



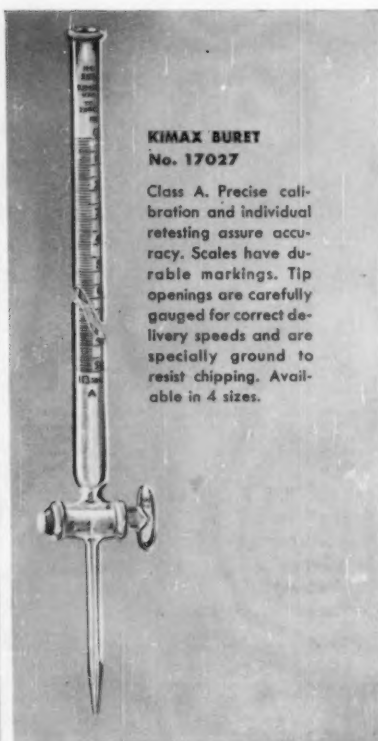
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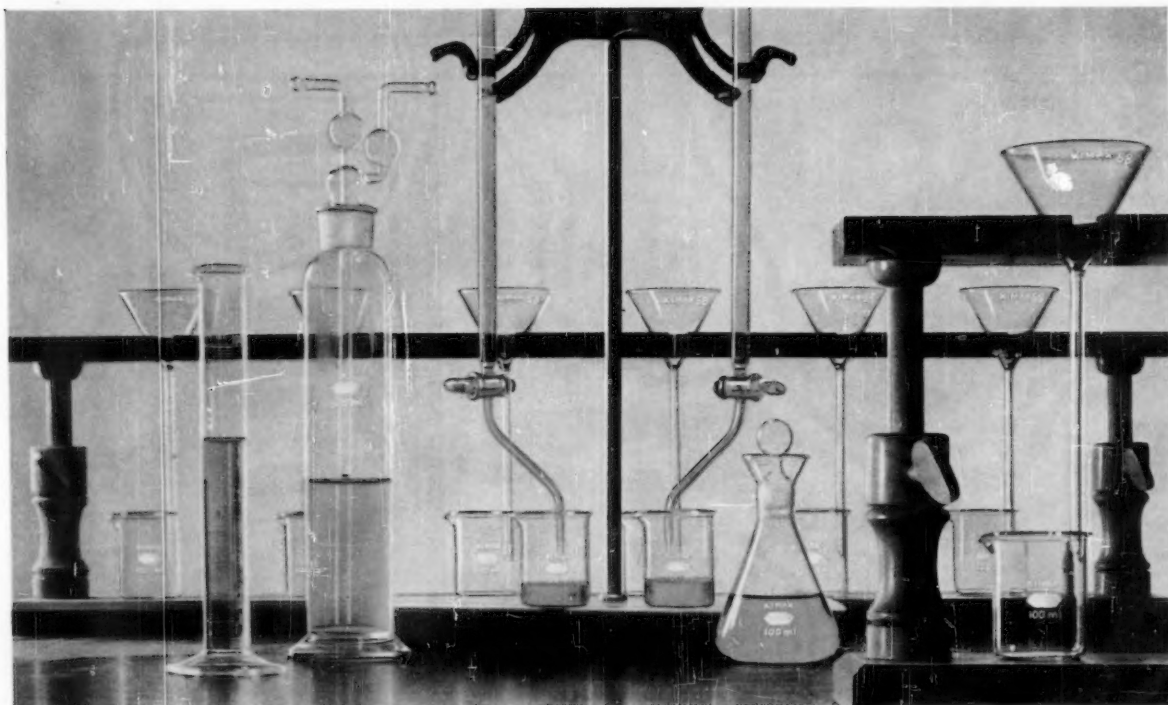
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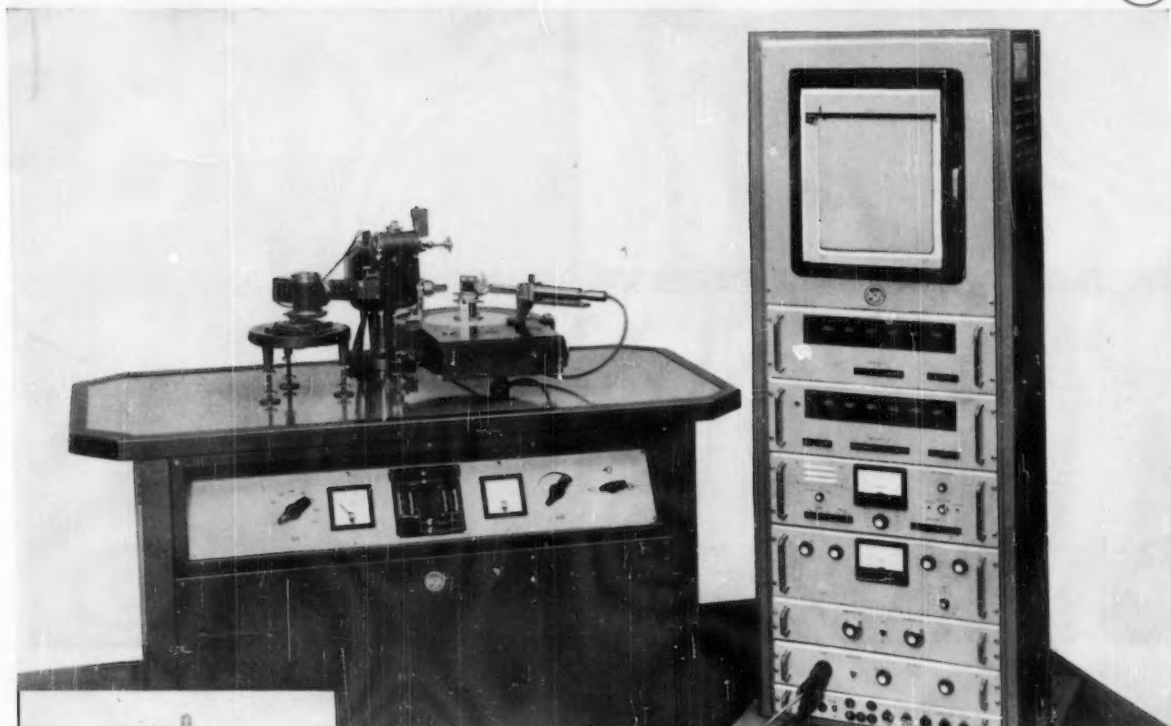
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ASTM BULLETIN

# ASTM Books in 1958-59

## Another Heavy Publication Year

THE Society's growth is reflected in the ever increasing volume of material published, and possibly no summary or statement of the Society's work is more effective than one covering the publications that are forthcoming.

The big 1958 Book of ASTM Standards—this year increased from seven to ten parts—and the special compilations of related standards are evidence of the growth in work and concentration on standardization of specifications and methods of testing. The special technical publications which contain the symposia and papers contribute greatly to that phase of the Society's work concerned with the promotion of the knowledge of materials.

Members will be advised of the appearance of these publications. As books become available, notices will appear in the ASTM BULLETIN. On most of the publications, special prices to members are in effect. Publications that are available are so indicated.

## Regular Publications . . .

### 1958 Book of ASTM Standards

Possibly the most important and certainly the biggest publication now in hand is the ten-part 1958 edition of the Book of ASTM Standards. This edition will total around 13,500 pages, 17 per cent larger than the 1955 edition. The ten volumes will be published as a grouping together of separate compilations. The ten parts are:

- Part 1 Ferrous Metals Specifications (Except Test Methods)
- Part 2 Non-Ferrous Metals Specifications (Except Test Methods), Electronics Materials
- Part 3 Methods of Test For Metals (Excluding Chemical Analysis)
- Part 4 Cement, Concrete, Mortars, Road Materials, Waterproofing, Soils
- Part 5 Masonry Products, Ceramics, Thermal Insulation, Sandwich and Building Constructions, Acoustical Materials, Fire Tests
- Part 6 Wood, Paper, Adhesives, Shipping Containers, Cellulose, Leather
- Part 7 Petroleum Products, Lubricants, Tank Measurements, Engine Tests
- Part 8 Paint, Naval Stores, Aromatic Hydrocarbons, Coal, Coke, Gaseous Fuels, Engine Antifreezes
- Part 9 Plastics, Electrical Insulation, Rubber, Carbon Black
- Part 10 Textiles, Soap, Water, Atmospheric Analysis, Wax Polishes



### Progress on Book of Standards

As this BULLETIN is being made up, presswork has been completed on Part 2, and books will be distributed to all whose requests are on file. This part is to be followed shortly by Parts 4 and 9, in that order. Work is now in progress on Parts 3, 10, 7, and 5 so that these parts can shortly go to press, looking toward their distribution before the end of the year. Parts 1, 6, and 8 will follow.

## Compilations of Standards

So many factors affect the release of the compilations that it is not possible at this time to give an accurate estimate of their size or the date they will become available. The size is affected by committee recommendations which may be submitted through the Administrative Committee on Standards, and the date of issue is governed somewhat by editorial considerations and the relation of these special compilations to the appearance of the various parts of the Book of Standards. The tabulations given below of special compilations therefore should be viewed as approximate.

### STANDARDS COMPILATION SCHEDULE

Sponsoring Committee	Title	Approximate Number of Pages	Approximate Appearance Date
A-1 . . . .	Steel Piping Materials	510	January
B-7 . . . .	Light Metals and Alloys (see p. 11)	344	October
B-8 . . . .	Electrodeposited Metallic Coatings	124	Available
C-1 . . . .	Cement (see p. 11)	276	October
C-9, D-4	Mineral Aggregates, Concrete, and Nonbituminous Highway Materials	384	October
D-2 . . . .	Petroleum Products and Lubricants	1160	December
D-3 . . . .	Gaseous Fuels	104	Available
D-4, D-8	Bituminous Materials for Highway Construction, Waterproofing and Roofing	488	November
D-7 . . . .	Wood	392	January
D-12 . . . .	Soaps and Other Detergents	180	December
D-13 . . . .	Textile Materials	880	November
D-20 . . . .	Plastics (see p. 11)	1096	October

## 1958 Index to ASTM Standards

This index provides the latest complete reference to the publications in which the various specifications and test methods appear. As the number of published ASTM standards becomes larger, the value and utility of the index increases.

## Year Book

The Year Book, which will be available shortly, contains a list of complete membership and official company-member representatives (name, title, address, company, etc.), personnel of all ASTM committees, geographical listing of the membership, as well as other useful information about the Society. It is furnished to members free of charge on request and may be purchased by committee members.

## 1958 Proceedings

The 1958 *Proceedings* will contain about 1500 pages of technical papers and discussion presented at the 1958 Annual Meeting. All reports submitted by the Society's technical committees at the Annual Meeting are also included.

The Gillett and Marburg Lectures are issued as separate publications and no longer appear in the *Proceedings*. Copies, however, are available without charge to members on request.

Following our usual practice, the papers and discussions presented at the Annual Meeting as part of special symposiums will appear in special technical publications.

## Special Technical Publications . . .

### Properties of Cast Iron at Elevated Temperatures (STP 248)

Sponsored by Steam Power Panel of ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals

An investigation was conducted on the properties of cast iron in the temperature range of 800 to 1000 F with the purpose of determining whether low-alloy cast iron can safely be used for load-carrying applications above the presently specified maximum of 650 F. The properties of six commercial low-gray cast irons and of one unalloyed ferritic nodular iron were evaluated at 800 and 1000 F by means of tension tests, creep-rupture tests, thermal-shock tests, growth tests, and metallographic examinations. The work which was begun in 1953 was done at the Southern Research Inst., Birmingham, Ala., under the direction of J. R. Kattus, head, Metallurgy Division. This report will be published as a special technical publication.

### High Temperature Metals—Their Role in the Technological Future—1958 Gillett Lecture\*

By Clyde Williams

The seventh Gillett Memorial Lecture was presented by Clyde Williams, president of Clyde Williams and Co., formerly president of Battelle Memorial Institute, on "High Temperature Metals—Their Role in the Technological Future." This lecture is jointly sponsored by ASTM and Battelle Memorial Institute and commemorates Horace W. Gillett, the first director of Battelle and one of this country's leading metallurgists.

The needs for materials for high-speed aircraft and missiles are discussed. Temperature problems in aircraft result from aero-

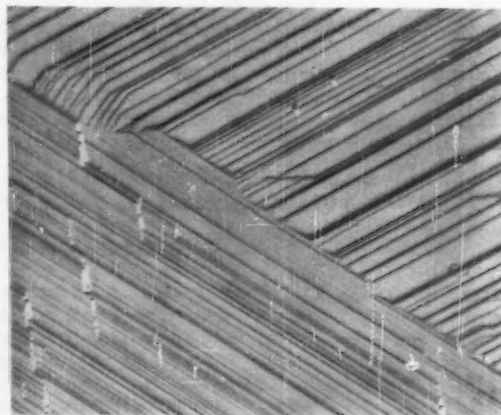
dynamic heating and from the nature of propulsion systems. Each level of speed, each type of engine, each fuel imposes special, and usually rigorous, materials problems. Progress is being made in the development of suitable temperature-resistance materials for airframes and skins, and in the development of truly "high-temperature" alloys and cermets for engine components. Cobalt, columbium, chromium, molybdenum, nickel, and tungsten appear to be the metals most likely to serve as the "workhorses" in the high-temperature age of flight and industrial processing. The demand for some of these metals could readily double or triple by 1965. Evidence exists that there are sufficient supplies of the ores of all these metals readily available to the United States to supply these increased amounts. Whatever the future demand, the Free World will be able to meet its need through technological development, economic adjustment, and planned programs of stockpiling, substitution, and conservation.

Since the Gillett Lecture is no longer included in the *Proceedings*, the lecture, with self covers, is available without charge to ASTM members on request. Additional copies to members (heavy paper cover) \$0.80, price to nonmembers \$1.00.

### Symposium on Basic Mechanisms of Fatigue (STP 237)\*

Sponsored by Committee E-9 on Fatigue

The general purpose of the symposium is to emphasize new observations of the basic mechanisms that are operative in metals subjected to repeated stressing. Much of the test data in the literature is of an applied nature. There is a strong need for understanding the fundamental readjustments taking place in the crystalline structure to evolve new or revised theory. Better concepts of the readjustments within slip systems and the correlation of these with the generation and movement of dislocations and vacancies are needed to assist in developing rational and practical methods of machine design. The papers in this symposium will clarify some of the mechanisms leading to fatigue damage and will help to emphasize the statistical nature of the material behavior under cyclic loadings. A variety of new observations made with the assistance of special techniques including photoelastic, damping, X-ray, and electron microscope studies are covered in the seven papers comprising the symposium volume.



Zirconium-Niobium [Columbium] Alloy

85 Weight per cent zirconium, 15 weight per cent columbium. Palladium preshadowed carbon replica. Original magnification: 3000 X; final magnification: 15000 X; reduced to one third size in reproduction.

First Prize, Electron Micrographs, Metals—Eleventh ASTM Photographic Exhibit W. H. Bridges, E. L. Long, Jr., and J. T. Houston, Oak Ridge National Laboratory, Oak Ridge, Tenn.

\* Presented at the 1958 Annual Meeting in Boston, Mass., June 22-27, 1958.



## Symposium on Solvent Extraction in the Analysis of Metals (STP 238)\*

Sponsored by Committee E-3 on Chemical Analysis of Metals

Great interest has been engendered in the past several decades in solvent extraction methods. Their simplicity, speed, convenience, and versatility have earned for extraction techniques a favored place among separation methods of interest to analysts. The purpose of this symposium is to better acquaint those interested with the applications of solvent extraction separation processes in the analysis of materials. Since solvent extraction procedures are only as good as the reagents used to produce extractable compounds, three of the symposium papers are devoted to detailed discussions of the characteristics and applications of three of the newer organic reagents—tri-*n*-octylphosphine, thenoyltrifluoroacetone, and 8-hydroxyquinoline. The basic principles involved in solvent extraction in ternary systems consisting of water, a mineral acid, and an organic solvent are covered in a fourth paper. The combination of organic solvent extraction and flame spectrophotometry presents advantages, and this is the subject of the final paper. Extraction and flame spectrophotometry should be attractive to laboratories considering emission spectrography but whose work loads, diversity of sample compositions, or cost factors have mitigated against its adoption.

## 1957 Supplement to the Bibliography and Abstracts on Electrical Contacts (STP 56-L)

Sponsored by Committee B-4 on Metallic Materials for Electric Heating, Electrical Resistance and Electrical Contacts—Prepared by Erle I. Shobert, II

This is the fifth supplement published to the 1952 edition. There has been a continuous growth of approximately 10 per cent per year in the size of the yearly supplement to this bibliography. This growth indicates that the basic scientific and technical work in this field is keeping pace with the developments in other fields of activity. The publication of this supplement makes it possible for everyone concerned with electrical contacts to keep currently up to date with the latest information. The subject index lists the same headings as were given in previous editions, namely, electric contacts—general; contact materials—fabricated; contact materials—powdered metals; circuit breaker design; circuit breaker testing; contactor or relay design; stationary or fixed contacts; sliding contacts—slip rings; sliding contacts—commutation; miscellaneous special applications; contact resistance; electric arc theory as applied to contacts; electric arc theory; glow discharge theory; spark discharge theory; contact wear; circuit and circuit parameters as applied to contact operation.

STP 56-L, 53 pages; heavy paper cover; price \$1.75; to members, \$1.40.

## Symposium on Cleaning of Electronic Device Components and Materials (STP 246)

Sponsored by Committee F-1 on Materials for Electron Tubes and Semiconductor Devices

The technology of designing and building electronic devices such as electron tubes and transistors has developed rapidly during the past decade or two. With this development, considerable technological emphasis has shifted from studies of the intrinsic properties of the materials used, to studies of the environments in which they must operate. The life and reliability of many electronic devices now appear to be more dependent on the contaminating influences of the impurities associated with the materials of construction than on the inherent behavior



Demonstration of the Low Density of Aluminum Foam

First Prize, General, Black and White—Eleventh ASTM Photographic Exhibit. Richard M. Reese, Bjorksten Research Laboratories, Inc., Madison, Wis.

of the materials themselves. This symposium, consisting of twenty-two papers, is divided into four categories, namely, physical and organic contaminants; gaseous contaminants; chemically combined contaminants; and soluble contaminants. This symposium brings out that significant improvement in device performance can be obtained as a result of using careful cleaning processes.

## Symposium on Advances in Electron Metallography (STP 245)

Sponsored by Subcommittee XI on Electron Metallography of Metals of Committee E-4 on Metallography

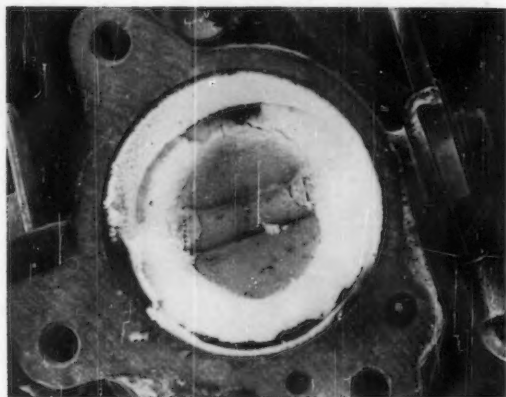
This symposium, consisting of nine papers, gives the latest information on advances in electron metallography. The subjects covered are: vibratory polishing of specimens for electron microscopy; a technique for the easy removal of "direct" replicas for electron microscopy; examination of metal foils by transmission electron microscopy; a study of dislocations in thin metal foils elongated in the electron microscope; microstructure of age hardenable alloys; an electron microscopic study of the precipitation hardening of commercial nickel-base alloys; the structure and composition of the  $\gamma'$  phase of commercial nickel-base alloys; electron probe analysis of segregation in Inconel; and the first progress report of the Non-Ferrous Task Group on metallography of titanium—8 per cent manganese alloy.

## ASTM Manual for Rating Diesel Fuels by Cetane Method

Sponsored by Committee D-2 on Petroleum Products and Lubricants

This new ASTM Manual, to be issued in 1959, will include the current Tentative Method of Test for Ignition Quality of Diesel Fuels by the Cetane Method (D 613 - 58 T) and also seven appendices which amplify the method and contain additional detailed information on the installation, operation, and maintenance of the Cetane Engine.

The first six appendices replace and bring up to date the information now furnished on the Cetane Method in Supplements I to VI to the 1952 ASTM Manual of Engine Test Methods for Rating Fuels. The new appendix VII describes in detail the ignition delay meter now used with the Cetane Engine.



**Ice Formation in Automobile Carburetor**  
*Eleventh ASTM Photographic Exhibit. Edmund F. Hawes, Universal Oil Products Co., Des Plaines, Ill.*

This is the last of three ASTM Manuals for Rating Fuels that have been in preparation. The other two are the 1956 Manual for Rating Motor Fuels by Motor and Research Methods, and the 1958 ASTM Manual for Rating Aviation Fuels by Supercharge and Aviation Methods. These three manuals replace the former 1952 Manual of Engine Test Methods for Rating Fuels.

### **Symposium on Stability of Distillate Fuel Oils (STP 224)**

**Sponsored by Technical Committee E on Burner Fuel Oils of Committee D-2 on Petroleum Products and Lubricants**

This symposium reviews the present situation with regard to the stability of home heating fuel oil and its effect on performance. The nine papers comprising the symposium help bring into focus the various types of stability problems, give some idea of their degree, and tell what has been, is being, or perhaps, should be done to relieve them. The subjects covered are: a review of the distillate fuel stability problem; how distillate fuel stability is measured and controlled; trials and tribulations of large development oil heating; do we need a stability specification for No. 2 heating oil; requirements for dependable performance of domestic oil burner nozzles; incompatibility of distillate fuels; distillate fuel oil gel; predictive type tests for storage stability and compatibility of diesel fuels; burning today's fuel oils.

### **Symposium on Effect of Water on Bituminous Paving Mixtures (STP 240)\***

**Sponsored by Committee D-4 on Road and Paving Materials**

Bituminous pavement failures are sometimes attributed to stripping of bitumen from the mineral aggregates due to a moist environment. Reasons for this unpredictable behavior are being sought by many investigators. Anti-stripping compounds to be added to the asphalt are being widely used. Observation of roads in service and the opinions expressed by observers are not entirely consistent, except that some asphalt-aggregate combinations appear to give better performance under moist conditions than others. The authors in this symposium discuss some of the latest thinking on the effect that each component, including environment, may contribute to the service behavior of bituminous paving mixtures and methods

that have been studied in an attempt to evaluate tests intended to predict such behavior. Field observations of the behavior of bituminous pavements as influenced by moisture are covered in one paper. Another paper reviews the important literature concerning the adhesion of bituminous materials to aggregates in the presence of water. The correlation of laboratory tests with field performance is an old subject, and one of the authors reviews a long-range program of correlation which involves the use of a laboratory circular track installation combined with a static immersion test. Additives, such as chemical agents, to aid in coating aggregates under adverse conditions have been commercially used for some time, and a study of nine commercial additives is discussed in one of the papers. In the last paper, the author reviews the proposed methods of test designed to measure or evaluate the resistance of bituminous paving mixtures to the deleterious effects of water which have varied from basic measurements of interfacial tension to simulated traffic tests.

### **Symposium on Applications of Soil Testing in Highway Design and Construction (STP 239)\***

**Sponsored by Committee D-18 on Soils for Engineering Purposes**

In recent years the expansion of the highway program has placed increased emphasis on the use of soil testing procedures both in design and construction. To present new developments in this field Committee D-18 arranged for a symposium of nine papers dealing with this phase of activity. The first paper covers the broad range of soil engineering problems as applied to highways. The second paper presents data pertaining to the use of power augers and earth-resistivity units to supplement drilling machines for highway exploration. The exploration of the banded sediments found in the difficult terrain along the Quebec, North Shore, and Labrador Railroad is covered in another paper. The soil exploration and mapping cooperative project in Illinois is described in one of the papers. One of the authors points out that the compaction of granular soils presents problems more difficult than other types and data were presented for several cohesionless soils using various methods of compaction. Another author describes the development of three typical sets of moisture-density curves used successfully for the control moisture and the compaction of soils in the construction of soil embankments by the Ohio Department of Highways. A soil classification scheme based on compaction tests is the subject of another paper. Also covered by one of the authors are laboratory shear tests on varved clays and their application to design. The final paper of the symposium presents the value of soil test data in local and regional road planning. It is felt that practicing engineers interested in highway design, those planning for new highways, and highway departments at both state and county level will find this symposium of value.

### **Symposium on Radiation Effects on Materials (STP 233)\***

**Sponsored jointly by ASTM Committee E-10 on Radioisotopes and Radiation Effects and the Atomic Industrial Forum**

The papers included in this symposium are designed to present as concisely as possible the major items of current interest in the field, and it is hoped that they will succeed, not only in making available new information, but in stimulating new ideas and plans for further work. The symposium is divided into three parts. The first one is on Dosimetry Techniques; the second on Irradiation Facilities and Techniques; and the third on Studies on Radiation Effects.

One paper is presented in the first category. Several do-

simetry techniques, such as the use of ion chambers, chemical dosimeters, etc., are compared under two conditions: pure gamma fields and reactor radiations. Also a review is made of some of the newer dosimetry techniques being investigated by various laboratories. In the second group, three different irradiation facilities are described and discussed. They are the NRU, a new, 200-megawatt research reactor at Chalk River in Canada; the Engineering Test Reactor (ETR) at the NRTS in Idaho, which has a design power of 175 megawatts; and a 5 megawatt reactor facility for the Sandia Corporation in Albuquerque, N. Mex. A paper is also presented on an automatic apparatus for in-pile fatigue testing. The third category is concerned with radiation effects. In general, the papers deal with new data concerning radiation effects to reactor materials and various other materials. In all, twelve papers comprise the symposium volume.

### **Symposium on Radioactivity in Industrial Water and Industrial Waste Water (STP 235)\***

**Sponsored by Committee D-19 on Industrial Water**

The advent of nuclear power has brought new problems to the field of water technology. In this symposium, problems in the reactor plant proper are discussed, the associated waste water processing and control methods described, and methods of analysis for radionuclides in industrial water and industrial waste water presented, including discussion of attendant radiation hazards. Also covered are the effects of nuclear operations on the environment and analysis of samples. The scope of the symposium was purposely broad enough to provide background information as to the conditions associated with the new phases of work on industrial water and industrial waste into which Committee D-19 is now moving actively. Purity of primary water must be at levels well beyond those previously required in order to minimize build-up of radioactivity and because of much greater problems of maintenance in the presence of radioactivity. It therefore becomes necessary to analyze for constituents in extremely low amounts, even smaller than parts per billion, for radioactive elements, for elements that change rapidly or disappear with time. Accordingly a whole new set of procedures for sampling and analysis must be developed. These problems are discussed. Also covered are methods for determining whether radiation hazards in water conform to standards for allowable limits. This is a timely symposium and shows the importance and impact of radioactivity on water analysis methods, and how water quality and analysis are of prime importance in the nuclear field.

### **Symposium on Paper and Paper Products—New Developments with Accompanying Requirements for Testing Methods (STP 241)\***

**Sponsored by Committee D-6 on Paper and Paper Products**

In the last few years the advances in the paper field have been especially rapid. These have included innovations in the paper-making process, in fiber composition, and in after-treatments of the finished sheet. This first ASTM symposium on new developments in paper will cover a few of the outstanding accomplishments with special consideration of the methods required for the testing of these new products. Seven papers are included in the symposium volume. An historical account of paper testing from the earliest weight tests, to radioisotope beta-ray thickness gages and identification of fibers by the electron microscope is covered in the first paper. Data on the effect that synthetic resins have on the internal bonding of paper and the modifications of existing test methods which may be needed in order to obtain reliable test data are presented

in the second paper. Another paper describes the technology and end uses of nonwoven fabrics and synthetic fiber papers. Testing of synthetic fiber papers is the subject of one of the papers. It is pointed out in another paper that hydroxyethylated cotton linters are providing the paper industry with a new furnish which will improve the characteristics of finished paper. Another paper reviews the testing program used to evaluate the characteristics of a new paper in which a large amount of carefully controlled machine direction stretch is built into the paper. The final paper deals with relative humidity measurements in packaging material testing, and the development of dew-point apparatus capable of precise measurements of dew-points in atmospheres at temperatures above ambient is described.

### **International Symposium on Plastics Testing and Standardization (STP 247)**

**Sponsored by ASTM (Committee D-20 on Plastics) for the American Group for ISO/TC-61**

Four main sections make up this symposium volume, which includes papers from Belgium, France, Germany, Hungary, The Netherlands, Poland, Sweden, Switzerland, Rumania, Russia, United Kingdom, and the United States. The first section is on How National Standards are Achieved, with most of the above mentioned countries contributing papers. The second chapter deals with Methods of Test for Engineering Properties of Plastics, and the subjects covered in this section are: basic considerations on the mechanical properties of molded plastic objects; anisotropic effects in testing plastics; most suitable shape for plastics materials specimens intended for measurement of their shear strength; encyclopedia of insulating materials; and mechanical properties of plastics at high speeds of testing. The third section covers Thermal Properties of Plastics and includes three papers on testing flow properties of thermosetting and thermoplastic molding compounds; stress-relaxation in polystyrene; and the Vicat softening point test method—correlations, users, and variants. The final section on Methods for Molecular Characterization consists of five papers covering the determination of accurate molecular weight of macromolecules through the measurement of dilute solution properties; the use of infrared spectroscopy in characterization of polymer structure; the use of X-ray diffraction and scattering in characterization of polymer structure; nuclear resonance studies of polymer chain flexibility; and suggestion for classification of macromolecular products based on the correlation between structure and properties of high polymers.

### **Symposium on Effect of Ozone on Rubber (STP 229)**

**Sponsored by Committee D-11 on Rubber-Like Materials**

Since World War II, there has been effective stimulation from at least two large consumers of rubber goods, namely, the United States Government and the automotive industry, for better ozone-resistant elastomeric polymers. The United States Government stores rubber parts all over the world and has parts in service on the earth's surface and in the stratosphere—all of which conditions cover a wide spectrum of ozone concentrations. The automotive industry has been vitally interested in the ozone resistance of rubber parts, both before and after the automobile moves into the customer's hands.

In addition to normal concentrations of ozone on the earth's surface, the climatic condition which has affected the atmosphere in the Los Angeles area, where both Government and the automotive industry have interests in rubber performance, has done much to stimulate interest in compounds to protect rub-



ber against the ravages of ozone and other deleterious gases in the atmosphere. Today, we do not have the complete answer to the problem, but the papers of the present symposium listed below show that progress is being made.

Rubber and Its Environment—A. J. Haagen-Smit  
Study of the Reaction of Ozone with Polybutadiene Rubbers—E.

R. Erickson and R. A. Berntsen

The Reaction of Ozone with Rubber—Harold Tucker

Ozone Resistance of Elastomeric Vulcanizates—Z. T. Ossefort

Chemical Antiozonants and Factors Affecting Their Utility—W. L. Cox

Prevention of Ozone Attack on Rubber by Use of Waxes—S. W.

Ferris, S. S. Kurtz, Jr., and J. S. Sweely

Comparison of Accelerated and Natural Tests for Ozone Resistance of Elastomers—G. N. Vacca

Quantitative Measurement of Rate of Ozone Cracking—A. G. Veith

Report on Interlaboratory Ozone Test Program of ASTM Committee D-11, Subcommittee XV, 1957.

STP 229, 136 pages; hard cover; price \$3.75; to members, \$3.00.

## Symposium on Some Approaches to Durability in Structures (STP 236)\*

Sponsored by Committee E-6 on Methods of Testing Building Constructions

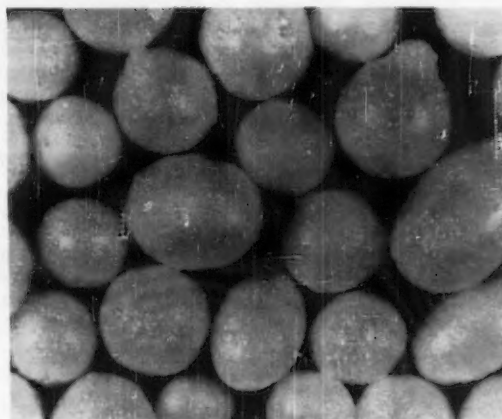
Durability in structures has received consideration by both the buyer and the builder for many years. Progress in improving structures from the standpoint of cost to give expected service has necessitated new engineering approaches which could predict service life. The older materials and techniques had service records which appealed to conservative buyers, but growing costs encouraged consideration of new approaches to building techniques. In evaluating these, durability, as a part of safety for people and property, has become of prime importance. Correlations between laboratory tests and field experience show no great agreement in engineering circles, particularly over the significance of test results, even if the competitive sales aspects are discounted. The five papers comprising this symposium are directed toward correlations between tests of some commonly used structural materials and the effects of weather on them, in the hope that future methods of test for durability might have more general acceptance among structural engineers and code authorities.

The subjects covered are: some factors affecting durability of structural clay product masonry, laboratory testing and the durability of concrete, durability tests of structural sandwich constructions, the durability of buildings—a discussion of some general aspects, effect of the atmosphere on masonry and related materials, and relation between actual and artificial weathering.

## Symposium on Particle Size Measurement (STP 234)\*

Sponsored by Committee E-1 on Methods of Testing

The purpose of this symposium is to bring up to date the earlier symposium held in 1941 entitled *New Methods for Particle Size Determination in the Sub-Sieve Range* (STP 51). Since that time a number of new techniques have been developed, some of which involved new types of apparatus for particle size measurement. These developments in instrumentation and technology are covered in the symposium papers. The symposium has three main categories consisting of sieves, sieving methods, and microscopy; sedimentation methods; and other particle size methods and correlations. Five papers comprise the section on sieves, sieving methods, and microscopy with papers on the mechanics of fine sieving, review of sieve



Prepuffed Expandable Polystyrene Bead

First Prize, General, Plastics—Eleventh ASTM Photographic Exhibit. Ruth Giuffria, General Electric Co., Louisville, Ky.

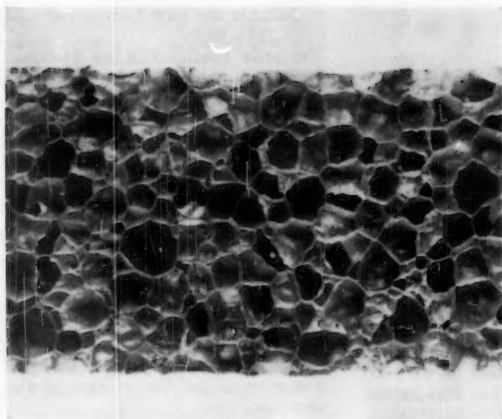
standardization, electroformed precision micromesh sieves, fluid cracking catalysts, and the particle bank of the Stanford Research Inst. Six papers comprise the category on sedimentation methods and cover recent developments in the hydrometer method as applied to soils, sedimentation procedures for determining particle size distribution, a photoelectric sedimentation method for particle size determination in the sub-sieve range, centrifuge sedimentation size analysis of membrane filter collected samples of airborne dusts, a liquid sedimentation method, and new methods for determining particle size distribution by examining gravitational and centrifugal sedimentation. In the last category on other particle size methods and correlations, five papers are included covering a discussion of the recommended practice of reporting particle size characteristics of pigments, determination of particle size by adsorption methods, a study of the Blaine fineness tester, three-dimensional electronic sizing of sub-sieve particles and turbidimetric particle size distribution theory.

## Symposium on Bulk Sampling (STP 242)\*

Sponsored by Committee E-11 on Quality Control in Cooperation with the Chemical Division of the American Society for Quality Control

The main purpose of the symposium is to provide further understanding of sampling on the part of those persons charged with the responsibility of preparing specifications involving sampling. Another objective is to give persons, faced with the interpretation of specifications and results based on sampling, a few rules to help prevent gross mistakes in making decisions. All too often the purposes and the consequences of the recommended sampling procedure are not clearly thought out, and for this reason the results may be misinterpreted in terms of the specifications. There is clearly a need for better planning in the preliminary stages of the materials' testing program before the specifications are set.

The problems related to sampling coal are discussed in two of the papers; another paper deals with measurement error consideration; and the final paper advises that model hypothesis, experiment, test, and development of a new hypothesis is vital to the solution of a bulk-sampling problem.



Lightweight Plastic Sheet with Pores

*Eleventh ASTM Photographic Exhibit.* L. M. Dogliotti, Quartermaster Research and Engineering Center, Natick, Mass.

### Index to the Literature on Spectrochemical Analysis, Part IV (1951-1955) (STP 41-D)

This is the fourth part of a series of bibliographical surveys of the literature of spectrochemical analysis. The first part (second edition) was published in 1941 and covered the literature for the years 1920 through 1939 with 1467 references. The second part, published in 1947, included about 1044 references (with detailed abstracts) to articles appearing in 1940 through 1945. Part III contains 1264 references and covers the years 1946 through 1950. It is expected that Part IV will also contain over 1000 references and will bring the bibliographical survey of the literature of spectrochemical analysis up to date through the year 1955.

### Man and Raw Materials—1958 Marburg Lecture\*

By Elmer Walter Pehrson

The Edgar Marburg Lecture was established as a memorial to the first secretary of the Society with the purpose of emphasizing the importance of furthering knowledge of properties and tests of engineering materials. At the 1958 Annual Meeting, the thirty-second Edgar Marburg Lecture entitled "Man and Raw Materials" was presented by Elmer Walter Pehrson, chief, Division of Foreign Activities, U. S. Bureau of Mines.

Mr. Pehrson discusses the fact that in the light of expanding world population and knowledge that mineral deposits are irreplaceable, recurring shortages understandably cause concern over the adequacy of material resources for the future. These anxieties are aggravated by the threat to military security implicit in potential shortages because raw materials are as indispensable in modern warfare as they are in maintaining peacetime prosperity. The lecture covers major political, economic, and technological factors affecting raw-material availability; the geopolitical implications of raw-material resources and production; and the problems confronting industrial nations in their access to raw-material resources in the backward areas of the world. Consideration is given to national and international raw-material policies and the industrial and military power inherent in the resource and potential raw-material strength of the Sino-Soviet Bloc, as well as a contrast of the resource limitations on industrial growth in the United States and the U.S.S.R.

Since the Marburg Lecture is no longer included in the *Proceedings*, the lecture, with self covers, is available free of charge to ASTM members on request.

## Now Available

## Compilations of Standards

### Light Metals and Alloys (B-7)

The new compilation of ASTM Standards on Light Metals and Alloys, will include the new color coding system for aluminum-base ingots as part of the Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179). Revisions have been made in 25 of the standards covering cast and wrought aluminum and magnesium, and their alloys as well as those for aluminum wire and cable, and light-metal die-casting alloys.

Also included are the new Specification for Aluminum Alloy Hardeners Used in Making Zinc-Base Die-Casting Alloys (B 327), and Aluminum Association alloy numbers in 9 newly revised specifications.

*Compilation of ASTM Standards on Light Metals and Alloys, Cast and Wrought;* 344 pages; paper cover; price \$4; to members, \$3.20.

### Cement (C-1)

Of major assistance to the national highway construction program and to the use of cement in concrete structures is the new compilation of ASTM Standards on Cement. The general refinement of standards by Committee C-1 on Cement is evidenced by the up-dating of 24 of the 34 standards included. Of particular interest are the revisions in the Specifications for Portland Blast-Furnace Slag Cement (C 205) and the Methods of Sampling Hydraulic Cement (C 183).

In addition to the standards, the compilation contains detailed information designed to assist the plant chemist in the performance of the cement tests.

*Compilation of ASTM Standards on Cement;* 272 pages; paper cover; price \$3.50; to members, \$2.80.

### Plastics (D-20)

In its 22-year history Committee D-20 on Plastics has developed a large number of standard test methods and specifications widely used by the plastics industry in research, to classify plastic materials, and to serve as a common language between producer and consumer. A new edition of the book "ASTM Standards on Plastics" now available, represents probably the most significant advance of any new edition of this well-known book. Of the 191 standards included, 43 were not included in the previous edition. Of these, 24 have been newly developed by Committee D-20 since the previous edition in 1957.

New material includes a group of standards on plastic pipe with specifications covering dimensions of SWP and IPS sizes for cellulose acetate butyrate, and acrylonitrile-butadiene-styrene pipe. Also included are methods for short- and long-term hydrostatic pressure tests of plastic pipe. These pipe standards have been developed jointly by the ASTM and by the Society of the Plastics Industry.

Also included in this new edition are tentative abbreviations of terms relating to plastics (D 1600). These new abbreviations have been developed in an effort to standardize a shorthand notation for some of the more generally used complicated chemical names. For example: acrylonitrile-butadiene-styrene plastics in the new notation are simply ABS plastics.

*Compilation of ASTM Standards on Plastics;* 1108 pages; paper cover; price \$8; to members, \$6.40.

# Actions on Standards

The Administrative Committee on Standards is empowered to pass on proposed new tentatives and revisions of existing tentatives, and tentative revisions of standards, as well as withdrawals of standards and tentatives offered between Annual Meetings of the Society. On September 9, 1958, the Standards Committee took these actions:

## Steel

**Tentative Specifications for Austenitic Steel Forged and Bored Pipe for High-Temperature Service (A 430 - 58 T)**

**New Tentative.**—These specifications cover four grades of heavy wall forged and bored austenitic steel pipe intended for high-temperature Central Station service. Pipe ordered under these specifications shall be suitable for bending and other forming operations and for fusion welding. Selection will depend on design, service conditions, mechanical properties, and high-temperature characteristics.

**Tentative Specification for High Strength Billet-Steel Bars for Concrete Reinforcement (A 431 - 58 T)**

**New Tentative.**—This specification covers deformed bars, rolled from new billet steel. The material is of a special high-strength grade and is intended for use in concrete structures designed for steel reinforcement with a high yield point.

**Tentative Specification for Carbon and Alloy-Steel Forgings for Turbine Generator Rotors and Shafts (A 292 - 57 T)**

**Tentative Specifications for Carbon and Alloy-Steel Forgings for Turbine Rotors and Shafts (A 793 - 57 T)**

**Revision.**—These revisions reflect the thoughts to date of a task group of Committee A-1 which has been considering the requirements necessary to insure quality in turbine and generator rotor forgings. There will probably be other revisions within the next few years as the task group continues its project.

**Tentative Specification for Welded Steel Wire Fabric for Concrete Reinforcement (A 185 - 56 T)**

**Revision.**—Revised to include galvanized fabric, and the procedures for tension tests and weld shear tests have been more fully described.

**Tentative Specification for Cold-Rolled Carbon-Steel Sheets for Deep-Drawn Parts (A 365 - 57 T)**

**Tentative Specification for Cold-Rolled Carbon-Steel Sheets, Commercial Quality (A 366 - 57 T)**

**Revision.**—Tolerances for sheets ordered to weight have been added.

**Tentative Specification for Flat-Rolled Carbon-Steel Sheets of Structural Quality (A 245 - 57 T)**

**Tentative Specification for Carbon-Steel Sheets for Flange and Firebox Qualities (A 414 - 57 T)**

**Revision.**—The tolerances for ordered width in Specification A 414 and for ordered weight and width in Specification A 245 have been revised in accordance with present commercial practice.

**Tentative Specification for Hot-Rolled Carbon-Steel Sheets, Commercial Quality (A 415 - 57 T)**

**Revision.**—Tolerances for sheets ordered to weight have been added, and the present width tolerances have been revised.

**Tentative Specifications for Billet-Steel Bars for Concrete Reinforcement (A 15 - 57 T)**

**Tentative Specifications for Cold-Rolled Carbon-Steel Strip (A 109 - 58 T)**

**Tentative Specifications for Flat-Rolled Carbon-Steel Sheets of Structural Quality (A 245 - 57 T)**

**Tentative Specification for Hot-Rolled Carbon-Steel Strip of Structural Quality (A 303 - 58 T)**

**Tentative Specifications for Cold-Rolled Carbon-Steel Sheets, Special Killed for Miscellaneous Drawn or Severely Formed Parts (A 365 - 57 T)**

**Tentative Specifications for Cold-Rolled Carbon-Steel Sheets, Commercial Quality (A 366 - 57 T)**

**Tentative Specification for High Strength Low Alloy Cold-Rolled Steel Sheets and Strip (A 374 - 54 T)**

**Tentative Specifications for High Strength Low Alloy Hot-Rolled Steel Sheets and Strip (A 375 - 54 T)**

**Tentative Specifications for Carbon Steel Sheets of Flange and Firebox Qualities (A 414 - 57 T)**

**Tentative Specifications for Hot-Rolled Carbon-Steel Sheets, Commercial Quality (A 415 - 57 T)**

**Tentative Specifications for Steel Sheets for Porcelain Enameling (A 424 - 58 T)**

**Tentative Specifications for Hot-Rolled Carbon-Steel Strip, Commercial Quality (A 425 - 58 T)**

**Tentative Specifications for Special Large Size Deformed Billet-Steel Bars for Concrete Reinforcement (A 408 - 57 T)**

**Revision.**—Revised to include the basic oxygen steelmaking process.

**Standard Specification for Cold-Drawn Steel Wire for Concrete Reinforcement (A 82 - 34)**

**Revision and Reversion to Tentative.**—Revised to include the basic oxygen process of steelmaking and to cover galvanized wire.

**Tentative Specification for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 - 57 T)**

**Revision.**—New low-carbon grade steels F304 L and F316 L have been added as well as a new grade F21 which has high temperature application. The heat treatment and mechanical property requirements have been revised.

**Tentative Specification for Forged or Rolled Carbon and Alloy Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 - 57 T)**

**Tentative Specification for Ferritic Steel Castings for Pressure Containing Parts**

**Suitable for Low-Temperature Service (A 352 - 58 T)**

**Tentative Specification for Seamless and Welded Steel Pipe for Low Temperature Service (A 333 - 57 T)**

**Revision.**—At the request of the ASME Boiler and Pressure Vessel Code Committee, provision has been made in these specifications for testing at the intended minimum operating temperature rather than requiring undercooling.

**Tentative Specification for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 - 58 T)**

**Revision.**—In Section 9 on Impact Properties, there has been added to the end of paragraph (a) the phrase "and shall be controlled within  $\pm 3$  F." This provides a control of the temperature of the test specimen.

**Tentative Specification for Carbon Steel Castings Suitable for Fusion Welding for High Temperature Service (A 216 - 57 T)**

**Revision.**—A new Section 18 on Repair of Defects has been added.

**Tentative Specification for Steel Castings for High-Temperature Service (A 351 - 57 T)**

**Revision.**—A new grade CH8 of austenitic steel has been added. It represents a modification of grade CH10 in the interest of improved weldability.

**Tentative Specification for Alloy Steel Castings for Pressure Containing Parts for High Pressure Service (A 217 - 57 T)**

**Revision.**—In Section 19 on Repair of Defects, the last sentence of Paragraph (b) has been deleted which reads "Heat treatments after weld repair other than tempering shall be performed only when agreed upon between the manufacturer and the purchaser." This sentence had been found to create confusion in the purchase of material to the specification.

**Tentative Specification for Factory-Made Wrought Carbon Steel and Ferritic Alloy Steel Welding Fittings (A 234 - 58 T)**

**Tentative Specification for Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 - 58 T)**

**Revision.**—The section covering hydrostatic testing has been revised to outline more clearly the present commercial practice.

**Tentative Specification for Seamless and Welded Steel Pipe for Low Temperature Service (A 333 - 57 T)**

**Revision.**—The chemical requirements of grade C has been changed to correlate with grade B of the Specification for Seamless Carbon Steel Pipe for High Temperature Service (A 106).

**Tentative Specification for Seamless Carbon Steel Pipe for High-Temperature Service (A 106 - 55 T)**

**Revision.**—Explanatory Note 2 has been changed to read as follows, the revision being the addition of the third sentence:

NOTE 2.—Grade A rather than grade B pipe should be used for close coiling, cold bending, or for forge welding. The purpose for which the pipe is to be used should be stated in the order. This note is not intended to prohibit the cold bending of grade B seamless pipe.



**Standard Specification for Carbon-Steel Blooms, Billets, and Slabs for Forgings (A 273 - 54)**

**Standard Specification for Alloy-Steel Blooms, Billets, and Slabs for Forgings (A 274 - 54)**

**Revision and Reversion to Tentative.**—The requirements in the chemical composition tables have been changed to agree with the AISI Manual covering alloy steel issued in July, 1955, and the AISI Manual covering carbon steel issued in July, 1957.

## Wrought Iron

**Tentative Specification for Wrought Iron Crane Chain (A 56 - 56 T)**

**Revision.**—The fourth sentence of Section 8(c) has been changed to read, "As soon as the beam drops, or the gage recedes from the ultimate load, the load is to be reduced to the seating load." This change is prompted by the identical change having been made in the Specification for Alloy Steel Chain (A 391 - 55 T).

## Corrosion of Iron and Steel

**Tentative Specifications for Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils, and Cut Lengths (A 93 - 58 T)**

**Tentative Specifications for Long Terme Iron or Steel Sheets, Coils, and Cut Lengths (A 308 - 58 T)**

**Tentative Specification for 1.25-oz Class Coating (Pot Yield) Zinc-Coated (Galvanized) Iron or Steel Roofing Sheets (A 361 - 58 T)**

**Revision.**—Recently there has been a change in industry practice as regards the permissible variations in tolerances from specified gage weight in sheet and strip production. In order that current commercial production operations be reflected, revisions have been made in Table IV of Specifications A 93, in Tables II and IV of Specifications A 308, and in Table III of Specification A 361.

## Copper and Copper Alloys, Cast and Wrought

**Tentative Specification for Rectangular Copper Wire for General Purposes (B 272 - 52 T)**

**Revision.**—This specification has been made complete within itself rather than referring for certain requirements to the Specifications for General Requirements for Wrought Copper and Copper-Alloy Wire (B 250 - 55 T). The title has been changed to read "Specification for Flat Copper Products with Finished (Rolled or Drawn) Edges (Flat Wire and Strip)."

**Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Wire (B 250 - 58 T)**

**Revision.**—There have been removed certain requirements relating to Specification B 272 which are no longer needed in view of the revision of B 272 mentioned above. The title has been changed to read "General Requirements for Wrought Copper-Alloy Wire."

## Thermal Insulating Materials

**Tentative Specification for Mineral Wool Molded-Type Pipe Insulation for Elevated Temperatures (C 281 - 52 T)**

**Revision.**—A complete revision of the specification was made necessary due to changes in manufacturing procedures and end uses for products qualified under this specification.

## Asbestos Cement Products

**Standard Specifications and Methods of Test for Corrugated Asbestos-Cement Sheets (C 221 - 55)**

**Revision and Reversion to Tentative.**—Revised by the addition of the new lightweight corrugated asbestos-cement sheets, and a change in the minimum breaking load in the standard corrugated values.

**Standard Specifications and Methods of Test for Flat Asbestos-Cement Sheets (C 220 - 55)**

**Standard Specifications and Methods of Test for Asbestos-Cement Roofing Shingles (C 222 - 55)**

**Standard Specifications and Methods of Test for Asbestos-Cement Siding Shingles and Clapboards (C 223 - 55)**

**Tentative Revision.**—The section on methods of test for deflection has been clarified. In addition, Section 4(b) of C 223, which refers to clapboards which are usually 4 ft in length, has been revised to permit shipment of shorter lengths in multiples of 16 in.

## Clay Pipe

**Tentative Specification for Jointing Vitri-fied Clay Pipe (C 425 - 58 T)**

**New Tentative.**—For many years there have been specifications for clay pipe but the composition and manner of jointing the pipe was left to the suppliers of numerous patented joint materials and processes. This standard for jointing clay pipe will enable the engineer to demand, by ASTM reference, standards of quality for the joints as well as the pipe.

**Standard Recommended Practice for Installing Vitri-fied Clay Sewer Pipe (C 12 - 54)**

**Revision and Reversion to Tentative.** This recommended practice has been brought into accord with present-day recognized and recommended good construction practices.

## Manufactured Masonry Units

**Tentative Method of Test for Determining the Moisture Condition of Hardened Concrete by the Relative Humidity Method (C 427 - 58 T)**

**New Tentative.**—The method based on relative humidity, provides an accurate indication of the moisture content of the concrete such as is needed to show whether it is at the desired degree of dryness to reduce drying shrinkage and cracking in the wall.

**Tentative Method for Determining the Drying Shrinkage of Concrete Block (C 426 - 58 T)**

**New Tentative.**—Drying shrinkage has

become an important item in present-day production of concrete block. This method of test is based on a modified British method which has found wide favor in Europe and in this country.

**Tentative Specifications for Ceramic Glazed Structural Clay Facing Tile, Facing Brick and Solid Masonry Units (C 126 - 55 T)**

**Revision.**—The revision consists of the addition of the 6M Series which is an American Standard modular size. Although it is currently being produced by only one manufacturer on the West Coast, it is generally considered to be the most popular size of glazed unit of this type in that area.

**Standard Specifications for Facing Brick (C 216 - 57)**

**Tentative Revision.**—This specification does not recognize surface colors, other than those resulting from sanding or flashing. In view of the increasing development and use of other types of surface colored facing brick, the following cautionary note has been added:

**NOTE.**—When surface colored brick, other than sanded or flashed, are specified for exterior use, the purchaser should require that data be submitted showing that after 50 cycles of freezing and thawing there is no observable difference in the applied finish when viewed from a distance of 10 ft under an illumination of not less than 50 ft-candles, by an observer with normal vision.

## Paint, Varnish, Lacquer and Related Products

**Tentative Method of Test for Ester Value of Lacquer Solvents and Diluents (D 1617 - 58 T)**

**New Tentative.**—This method covers a procedure for determining the ester value of specification grade lacquer solvents and diluents. The sample is reacted with a measured excess of aqueous potassium hydroxide using isopropanol as a mutual solvent if necessary. The amount of potassium hydroxide consumed, which is determined by titrating the excess with standard mineral acid, is a measure of the ester originally present. This method was published previously as Section 12 on Ester Value of the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 - 53).

**Tentative Method of Test for Melamine Content of Nitrogen Resins (D 1597 - 58 T)**

**New Tentative.**—This method covers the spectrophotometric determination of melamine in butylated or caprylated melamine-formaldehyde resins and in mixtures of such melamine and urea resins, but does not cover analogous resins made from substituted melamine derivatives.

## Petroleum Products and Lubricants

**Tentative Specifications for Gasoline (D 439 - 58 T)**

**Revision.**—The minimum research octane number for regular and premium grades of motor gasolines have been raised one unit.

**Tentative Method for Color of Petroleum Products (D 1500 - 57 T)**

**Revision.**—The tolerances for the standard color glass have been doubled since it was found that the previous tolerances were unduly restrictive and imposed increased costs on the consumer as a result of the difficulty in manufacture.

**Tentative Methods for Calibrating Tank Car Tanks (Pressure and Non-Pressure Types) (D 1409 - 56 T)**

**Withdrawal.**—This method has never been published since Committee D-2 has not agreed on the final text. Considerable work needs to be done before a final text is available, and rather than keep a method number on the books that is not supported with the method's test, it has been felt desirable to withdraw the method entirely.

## Industrial Aromatic Hydrocarbons and Related Materials

**Tentative Method of Test for Solidification Points of Chemicals (D 1493 - 57 T)**

**Revision.**—Difficulty had been experienced in connection with reporting of temperatures, specifically in connection with the rounding off of values, and Section 7 on Report has been changed to correct this difficulty.

## Soils for Engineering Purposes

**Tentative Definitions of Terms and Symbols Relating to Soil Mechanics (D 953 - 42 T)**

**Revision.**—This revision is the result of a comprehensive review of all terms which have been compiled relating to soil mechanics and foundation engineering by Committee D-18 and a special committee of the American Society of Civil Engineers. The previous definitions and symbols have been replaced with this revision.

## Plastics

**Tentative Specification for Dimensions of Iron Pipe Size (IPS) Extruded Acrylonitrile-Butadiene-Styrene (ABS) Plastic Pipe (D 1527 - 58 T)**

**New Tentative.**—This specification covers the requirements for the dimensions and tolerances of acrylonitrile-butadiene-styrene (ABS) plastic pipe conforming to standard iron pipe sizes (IPS). This pipe shall be extruded from a thermoplastic compound in which combinations of acrylonitrile, butadiene, and styrene or its derivatives are the principal components.

**Tentative Specification for Dimensions of Solvent Welded (SWP Size) Extruded Acrylonitrile-Butadiene-Styrene (ABS) Plastic Pipe (D 1528 - 58 T)**

**New Tentative.**—This specification covers the requirements for the dimensions and tolerances of plastic pipe designated as Solvent-Welded Pipe (SWP Size). This pipe shall be extruded from a thermoplastic compound in which combinations of acrylonitrile, butadiene, and styrene or its derivatives are the principal components. Acrylonitrile-butadiene-styrene

(ABS) plastic pipe of this size is intended to be joined together by solvent bonding only.

**Tentative Specification for Cellulose Propionate Molding and Extrusion Compounds (D 1562 - 58 T)**

**New Tentative.**—There is covered by this specification plasticized cellulose propionate thermoplastic compounds suitable for injection and extrusion molding, the cellulose propionate having a propionyl content of at least 38 per cent and an acetyl content of not more than 9 per cent. The compounds may or may not contain dyes and pigments. This cellulosic plastic, because of outstanding toughness and excellent finish when molded, is widely used for telephones, pens and pencils, appliance housings, steering wheels, knobs, etc.

## Carbon Black

**Tentative Method of Test for Discoloration of Benzene by Carbon Black (D 1618 - 58 T)**

**New Tentative.**—This method covers a procedure for determining the degree of discoloration of benzene by carbon black, by means of a photoelectric colorimeter or spectrophotometer. The results of this test indicate the degree to which rubber compounded with the black will cause staining of lacquers or will bleed color through white sidewalls of tires.

**Tentative Methods of Test for Sulfur Content of Carbon Black (D 1619 - 58 T)**

**New Tentative.**—There is covered procedures for determining the sulfur content of carbon black by the oxygen bomb method, and the combustion furnace method. As sulfur is a vulcanizing agent for rubber, it is desirable to know how much may be introduced into the rubber compound with the carbon black.

**Tentative Method of Test for Volatile Content of Carbon Black (D 1620 - 58 T)**

**New Tentative.**—This method covers a procedure for determining the volatile material other than moisture in carbon black. When carbon black is used as a pigment in paints and inks, the volatile content affects the flow; therefore for such application it is important to know the volatile content.

## Methods of Testing

**Tentative Method of Test for Brinell Hardness of Metallic Materials (E 10 - 54 T)**

**Revision.**—Generally revised to improve and clarify the test procedure.

**Tentative Definitions of Terms Relating to Methods of Mechanical Testing (E 6 - 58 T)**

**Revision.**—The revision consists of the addition of nine definitions relating to creep of materials.

**Standard Definitions of Terms Relating to Rheological Properties of Matter (E 24 - 42)**

**Tentative Revision.**—A number of definitions have been completely revised as the result of extensive study by a task group of all the definitions relating to rheology.

**Standard Specifications for Sieves for Testing Purposes (Wire Cloth Sieves, Round-Hole and Square-Hole Screens or Sieves) (E 11 - 39)**

**Tentative Revision.**—The present nominal permissible range for wire diameters for each sieve has been changed to a single recommended nominal wire diameter with a permissible deviation expressed in percentage for range of sieve opening.

## Materials for Electron Tubes and Semiconductor Devices

**Tentative Method of Test for Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps (F 205 - 56 T)**

**Revision.**—Changes in the density values in Table I were made to align this specification with the newly adopted densities in the Specification for Round Wire for Use as Grid Lateral Winding Wire in Electron Tube Grids (F 290 - 57 T).

**Tentative Method of Test for Sag of Tungsten Wire (F 269 - 52 T)**

**Revision.**—The size ranges of wire that may be tested under this specification have been increased, and the exposed test lengths and weights are included for these larger diameter sizes. The diameter of bend for the hairpin has been changed.

## Offers of Papers for 1959 Meetings

Annual Meeting, June 21-26, Atlantic City, N. J.

Third Pacific Area National Meeting, October 11-16, San Francisco, Calif.

THE Administrative Committee on Papers and Publications will meet early in 1959 to consider the papers to be published by the Society in 1959 and to develop the program for the Annual Meeting in June and the Third Pacific Area National Meeting in October.

All those who wish to offer papers for presentation at these meetings and for publication by the Society should send these offers to headquarters not later than January 1 for the Pacific Coast papers and January 10 for the Annual Meeting papers.

All offers should be accompanied by a summary which will make clear the intended scope of the paper and which will indicate features of the work that will, in the author's opinion, justify its publication and inclusion in the programs of the Annual Meeting or Pacific Area National Meeting.

Suitable forms for use in transmitting this information will be sent promptly upon request to headquarters.

By WALTER H. SMITH<sup>1</sup>

AT THE Society's Annual Meeting in Boston the technical program included a Symposium on Materials Research Frontiers. The purpose of this activity was to bring before the Society a brief compendium and discussion of information on the progress of research relating to basic knowledge and understanding of material properties. There were eight speakers, all of them recognized authorities in the respective fields. Although the talks extended through an entire day, the meeting room was constantly filled (and at times crowded) with an attentive and appreciative audience.

ACR, as a cosponsor of the symposium (with the New England District Council) is pleased, of course, that the undertaking was such a success. However, we mention the matter here for quite another reason. We believe that the symposium from every aspect served to emphasize the general and wholehearted recognition of the close relation of materials research to the interests and activities of ASTM. This is an important point. For there are still some—even members—who regard ASTM strictly as a testing and specifications society and fail to see where research applies. True, the establishment and maintenance of standards is a most important part of our work, but it is also an important objective of the society to promote the knowledge of materials. To accomplish either of these purposes requires the touch and influence of research in many ways.

Consider the preparation of standards. Specialized technical committees operate in this broad and detailed activity of the society. The members are drawn from every part of industry, from government agencies, technical institutions, and colleges. Whether realizing it or not, these carefully selected committee members bring into their working groups wisdom, experience, and knowledge gained from many studies in many places. Although a standard or method developed by the committee may seem simple and even obvious, it is based on the sound judgment of several able minds and has secure roots.

Sometimes the criticism is heard that there is no evidence of research relative to the development of a certain standard. In many cases it may not be possible to produce this evidence readily because

the contributing bits of data, knowledge, and intellectual skill have been brought together from multiple sources. Yet we can say with all assurance that every ASTM standard has research in its ancestry.

We must remember too that ASTM standards and publications, in turn, themselves contribute to research. The quest for new knowledge requires working tools in the form of special apparatus, experimental procedures, or methods of deriving information on the properties of a material. Often these have already been worked out by ASTM to the great convenience of the investigator.

As for the many excellent technical papers presented to and published by the Society, certainly they represent or are supported by research. And this

statement of course applies to the Marburg and Gillett Lecture presentations. For the advancement of knowledge of materials requires recognition both of past accomplishments and of work on the advancing front where new theories, information, and discoveries shape the form of things to come.

The Symposium on Materials Research Frontiers was something of an experiment. We believe that its excellent reception indicates acceptance of this kind of subject session by the Society membership. We were fortunate indeed to have such an outstanding and learned panel of speakers; we are grateful for the thought, time, and effort they gave to ASTM. Hopefully, the pattern and level of quality set by them will be continued by others.

### NBS Standard Samples of Phosphors

THE National Bureau of Standards has recently made available 14 standard samples of phosphors (see table below) selected in cooperation with the Electrochemical Society. The main concern in preparing the samples was to achieve uniformity of characteristics and thus to provide a fixed basis for comparison with other phosphors.

As the physical properties of these samples have not yet been measured, it is intended that they serve only as common reference materials for comparison of research phosphors. However, for the most part they represent the optimum efficiency of phosphors currently produced. An investigation of their basic physical properties is planned.

TABLE I.—PHOSPHOR STANDARD SAMPLES.

Sample	Name	Symbolic Formula	Remarks	Approximate Weight, g	Price per sample
No. 1020..	Zinc sulfide phosphor	ZnS:Ag	Blue component of P-4 phosphor	14	\$3.00
No. 1021..	Zinc silicate phosphor	Zn <sub>2</sub> SiO <sub>4</sub> :Mn	P-1 phosphor as used in cathode-ray tubes	28	3.00
No. 1022..	Zinc sulfide phosphor	ZnS:Cu	P-2 phosphor	14	3.00
No. 1023..	Zinc-cadmium sulfide phosphor	ZnCdS:Ag	Yellow component of P-4 phosphor	14	3.00
No. 1024..	Zinc-cadmium sulfide phosphor	ZnCdS:Ag	Orange component of P-14 phosphor	14	3.00
No. 1025..	Zinc phosphate phosphor	Zn <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> :Mn	Red component of P-22 phosphor	28	3.00
No. 1026..	Calcium tungstate phosphor	CaWO <sub>4</sub> Pb	...	28	3.00
No. 1027..	Magnesium tungstate phosphor	MgWO <sub>4</sub>	...	28	3.00
No. 1028..	Zinc silicate phosphor	Zn <sub>2</sub> SiO <sub>4</sub> :Mn	As used in fluorescent lamps	28	3.00
No. 1029..	Calcium silicate phosphor	CaSiO <sub>3</sub> :Pb, Mn	...	14	3.00
No. 1030..	Magnesium arsenate phosphor	(MgO) <sub>2</sub> (As <sub>2</sub> O <sub>3</sub> ) <sub>3</sub> :Mn	...	28	3.00
No. 1031..	Calcium halophosphate phosphor	3Ca <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> ·Ca(F, Cl):Sb, Mn	...	28	3.00
No. 1032..	Barium silicate phosphor	BaSi <sub>2</sub> O <sub>6</sub> :Pb	Near ultraviolet emission	28	3.00
No. 1033..	Calcium phosphate phosphor	Ca <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> :Ti	Erythral ultraviolet emission	38	3.00

<sup>1</sup> Arthur D. Little, Inc., Cambridge, Mass.





OCTOBER 1958

NO. 233

NINETEEN-SIXTEEN  
RACE STREET  
PHILADELPHIA 3, PENNA.

## Staff Administrative and Personnel Changes

IN addition to two additions to the technical Staff, the responsibilities of two other Staff members have been clarified through new titles.

Albert L. Batik is named Advertising Manager and will continue his work with districts and in the development area. Henry J. Stremba becomes Junior Assistant Technical Secretary, recognizing his activities with technical committees, but will have continued and increasing responsibilities on publications, particularly involving standards. Both of these young men with engineering training, Mr. Batik's also in business administration, have been assuming more responsibility.

Recent additions include G. George Dochnahl who received his engineering education in Germany. He is currently concentrating on technical papers but also aiding in publications involving standards.

A. Q. Mowbray, the most recent addition, comes to us from the Hamilton Standard Division, United Aircraft. His engineering degree was obtained from University of Delaware, in mechanical engineering. Later he taught and received his Masters Degree in Theoretical and Applied Mechanics at the University of Illinois. His experience has included some years in publishing work. Currently he is aiding effectively in expediting publications involving standards and gradually will begin to follow certain other publications and technical aspects of the Society's work.

All four men are married, Mr. Mowbray having three children, Mr. Batik, the father of two, and Mr. Stremba having one.

## Little Book Gets Around

THE booklet "So You Are Now a Committee Member" recently distributed to all the members of the Society, as well as to committee members, has drawn a large number of favorable comments. Naturally this makes the Headquarters' Staff very proud as it indicates that the booklet is serving its intended purpose—to explain to the new member the how and why of technical committee operations.

The booklet has also drawn favorable comment from other societies and three of them have requested copies of the booklet in quantity for distribution to their members.

The Board of Directors and the Staff had long felt the need for some kind of assistance for the new member of a committee. Several of the larger committees had actually initiated programs of indoctrination for new members to explain the objectives, organization, and working procedures of the Society and of the committees. Such programs are very desirable as they can be designed specifically for a particular committee. Naturally, the detailed operations and procedures are not necessarily the same for all committees; the Society's Regulations provide only the policy framework within which the committees can work.

As for the preparation of the booklet itself, it was the product of many people. About five manuscript drafts and innumerable conferences within the Staff as well as a large stack of correspondence with reviewers preceded the submission of copy to the printer. Early drafts were improved by the many detailed comments and suggestions received from reviewers. Later drafts were sent for review to several Directors and to the Administrative Committee

on Standards. These comments and what had gone before provided material for a final draft. Then came conferences with the cartoonist-illustrator for the drawings of skinny-legged men whose resemblance to any ASTM members is purely unintentional.

We believe Lee DeGroot's drawings were a big factor in the success of the booklet. But more important was the team operation, the working together of the Staff and the many conscientious reviewers whose efforts contributed so importantly to the booklet's success. The Directors and Staff sincerely thank all who had a hand in it.

## National Conference on Air Pollution

THE National Conference on Air Pollution will be held at the Sheraton-Park Hotel in Washington, D. C. on November 18, 19, and 20. The interdisciplinary meeting has been called by Dr. Leroy E. Burney, Surgeon General, Public Health Service, U. S. Department of Health, Education, and Welfare.

Air pollution is becoming increasingly serious as population growth and industrial expansion add to the volume of pollutants discharged into the far-from-limitless ocean of air that surrounds us. Although the problem has recently received considerable attention from governmental bodies as well as from industries and voluntary organizations, there has been no opportunity for representatives of all these groups to meet together to assess their progress and to develop a coordinated plan for future action. This conference has been organized to meet this need.

## 1959 Award of Merit Committee

THE 1959 Award of Merit Committee, appointed by the Board of Directors, includes E. O. Hausman and A. A. Jones, serving with holdover members H. L. Maxwell, chairman, and L. C. Beard, Jr. R. T. Kropf will represent the Board of Directors.

In accordance with Rules Governing Award of Merit (see current Year Book) all suggestions for 1959 Awards must be in the hands of the executive secretary of the Society for consideration by the Award of Merit Committee not later than February 1, 1959.

## Physical Metallurgy of Stress-Corrosion Fracture

THE American Institute of Mining, Metallurgical, and Petroleum Engineers has announced that a conference on the Physical Metallurgy of Stress-Corrosion Fracture will be held April 2-3, 1959 at the Mellon Institute, Pittsburgh, Pa. ASTM, the Electrochemical Society, and the National Association of Corrosion Engineers are cooperating with the Metallurgical Society of AIME in the technical program.

The program is intended to provide a broad basis for presentation and discussion of recent fundamental advances in stress-corrosion cracking. Session topics will include theoretical aspects of stress corrosion and fracture; new experimental advances in stress corrosion and fracture; and mechanisms of stress-corrosion cracking, including environmental and metallurgical aspects and the behavior of specific materials. Emphasis will be placed on the physical metallurgical, surface-chemical, and solid state physics aspects of the phenomenon.

On April 2, the sessions, as presently planned, will take up:

### Introduction and Background:

- (1) significance of the stress-corrosion problem, and
- (2) characteristics of stress-corrosion cracking.

### Theoretical Aspects of Stress, Corrosion, and Fracture

- (1) relationship of microstress to fracture (at or near solid-fuel interfaces),
- (2) thermodynamic relationship between stress and chemical potential, and
- (3) relationship of surface topology and structure to environment.

### Experimental Aspects of Stress, Corrosion, and Fracture:

- (1) role of the corrosion product,
- (2) stress and microtopology, and
- (3) electrode potential.

On April 3 the program will include:

### Mechanisms of Stress-Corrosion Cracking:

- (1) environmental aspects,
- (2) metallurgical aspects, and
- (3) behavior of specific materials.

The sessions will be of the discussion type. Speakers will present summaries of their papers, which are to be pre-printed and circulated in advance, thus providing that most of the time of the conference may be devoted to discussion and exchange of scientific information.

## 1959 Electronic Components Conference

"New Concepts for the Space Age" is the theme of the 1959 Electronic Components Conference, sponsored by the American Institute of Electrical Engineers, Institute of Radio Engineers, Electronic Industries Assn., and West Coast Electronic Manufacturers Assn. The conference will be held at the Benjamin Franklin Hotel in Philadelphia, Pa., on May 6-8, 1959.

Conference topics will emphasize components and their applications in space vehicles, covering micro-miniaturization and the new component—the micro-module, the applications of new materials and new techniques, reliability,

and recent improvements in electronic components and passive components. The papers will range from new developments, applications, and fabrication techniques for transistors, diodes, and rectifiers to data handling components and instrumentation for electronic sensing of altitude, radiation, acceleration, temperature, etc. The theoretical aspects will also be treated. The future requirements for military and industrial applications will be projected and the effects of radiation on materials as well as transistors and vacuum tubes will receive particular attention.

## Schedule of ASTM Meetings

This gives the latest information available at ASTM Headquarters. Direct mail notices of all district and committee meetings customarily distributed by the officers of the respective groups should be the final source of information on dates and location of meetings. This schedule does not attempt to list all meetings of smaller sections and subgroups.

Date	Group	Place
Nov. 5-7	Committee E-13 on Absorption Spectroscopy	New York, N. Y. (Hotel New Yorker)
Nov. 7	Joint Committee on Chemical Analysis by Powder Diffraction Methods	Pittsburgh, Pa. (Mellon Inst.)
Nov. 10	Detroit District	Detroit, Mich. (Calvert Hall)
Nov. 11	Pittsburgh District	Pittsburgh, Pa. (Westinghouse Research Center)
Nov. 13-14	Committee F-1 on Materials for Electron Tubes and Semiconductor Devices	Sky Top, Pa. (The Inn)
Nov. 17	Central New York District	Troy, N. Y. (Rensselaer Polytechnic Inst.)
Nov. 19	Washington, D. C. District	Washington, D. C.
Nov. 19-22	Committee C-13 on Concrete Pipe	Chicago, Ill. (Union League Club)
Nov. 20	Philadelphia District	Villanova, Pa. (Villanova College)
Dec. 1-3	Committee C-1 on Cement	Lafayette, Ind. (Purdue Memorial Union)
Dec. 3	Chicago District	
Dec. 3-5	Committee C-9 on Concrete and Concrete Aggregates	
1959		
Jan. 14	Chicago District	Chicago, Ill.
Jan. 14-16	Committee A-1 on Steel	Chicago, Ill. (LaSalle Hotel)
Jan. 19-21	Committee D-1 on Paint, Varnish, Lacquer and Related Products	Columbus, Ohio (Deshler-Wallick Hotel)
Jan. 21	Washington, D. C. District	Raleigh, N. C. (N. C. State College)
Jan. 25-28	Committee D-19 on Industrial Water	Savannah, Ga. (General Oglethorpe Hotel)
Feb. 1-6	Committee D-2 on Petroleum Products and Lubricants	St. Louis, Mo. (Sheraton-Jefferson Hotel)
Feb. 2-6	Committee Week	Pittsburgh, Pa. (Penn-Sheraton Hotel)
Feb. 4-6	Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials	St. Louis, Mo. (Sheraton-Jefferson Hotel)
Feb. 25-27	Committee D-6 on Paper and Paper Products	New York, N. Y. (ASA Headquarters)
April 5-10	Fifth Nuclear Congress	Cleveland, Ohio (Shoreham Hotel)
April 13-14	Committee D-15 on Engine Antifreezes	Washington, D. C. (Atlantic City, N. J.)
June 21-26	Annual Meeting	Atlantic City, N. J. (Chaifonte-Haddon Hall)
Oct. 11-16	Third Pacific Area National Meeting	San Francisco, Calif. (Sheraton-Palace Hotel)

# District Activities

## SOUTHEAST and OHIO VALLEY

### President Tours Southeast

In a week-long tour, Professor Woods, ASTM President, accompanied by R. J. Painter, Executive Secretary, toured the Southeast and Ohio Valley Districts from October 6 to 10. The first meeting, Monday, October 6, held jointly with the Gainesville Section of the American Society for Civil Engineers and the Student Chapter of ASCE, was held at the University of Florida. Based on his long experiences with construction projects in Alaska, north-



President Woods

ern Canada, and Labrador, Professor Woods discussed "Polar Construction," devoting much of his talk to the effects of frost action and permafrost.

On Wednesday, October 8, at a meeting held in cooperation with the Georgia Section ASCE, the Georgia Engineering Society, the Society of American Military Engineers and the Georgia Section of ASME, Professor Woods discussed "Stretching Your Highway Dollar Through Research." The meeting was held at the Georgia Institute of Technology. Considerable assistance in planning the meeting was given by the Research Institute of GIT.

The following day, Thursday, October 9, Professor Woods spoke before a joint meeting of the Technical Society of Knoxville, the Knoxville Branch ASCE and Student Chapter ASCE, the Knoxville Chapter of the Tennessee

Society of Professional Engineers, and the Student Chapter of the American Society of Military Engineers. The meeting was held on the campus of the University of Tennessee. Once again the topic was "Polar Construction."

These meetings were all under the auspices of the Southeast District of ASTM.

Professor Woods and Mr. Painter then moved into the state of Kentucky to attend a joint meeting of the Ohio Valley District of ASTM and the Blue Grass Chapter of the Kentucky Society of Professional Engineers at the Phoenix Hotel in Lexington, Ky, Friday, October 10.

## PHILADELPHIA

### Sprague Speaks on "The Defense Program and Reliability"

At a joint meeting of the ASTM Philadelphia District and ASTM Committee F-1 on Materials for Electron Tubes and Semiconductor Devices, Julian B. Sprague, president of the Sprague Electric Co., spoke on "The Defense Program and Reliability." The dinner meeting, held in the Rose Room of McAllister's, was attended by leaders of industry and science from the electronics and missile component field. The dinner meeting was arranged in conjunction with a program planned by Committee F-1 which included a four-session, two-day symposium held in Philadelphia on the cleaning of electronic device components and materials. Frank J. Biondi, Bell Telephone Laboratories, headed the program committee for Committee F-1 and Philadelphia District arrangements were made under the direction of A. H. Kidder, chairman of the Philadelphia District.

## ST. LOUIS

### Structural Engineering Reviewed

At a joint meeting of the St. Louis District of ASTM, the St. Louis Chapter of the American Welding Society, and the Engineers Club of St. Louis, Dr. Wayne Teng, Chief Structural Engineer of the John F. Meissner Engineers, Inc., spoke on "New Dimensions in Structural Engineering." Dr. Teng reviewed recent developments and applications in structural engineering. He discussed new techniques, materials, and problems which confront structural engineers today. Heavy emphasis was placed on the application of structural welding. Arrangements for the meeting, held on the campus of Washington University, were made by W. C. Magruder, chairman of the ASTM St. Louis District.

### Alloy Data Requested

THE Data and Publications Panel of the ASTM-ASME Joint Committee on Effects of Temperature on the Properties of Metals has nearly completed a survey of physical properties of alloys at low and high temperatures.

Under this project, the panel has been soliciting physical property data over a 2-year period. Data forms and letters of solicitation were distributed to some 400 laboratories during this time.

The data accumulated are being evaluated for publication in the near future. Anyone who has not yet contributed data and wishes to do so should immediately write or call E. E. Reynolds, Allegheny Ludlum Steel Corp., Watervliet, N. Y.

## Coming District Meeting

District	Program
New England	President Woods speaks on "Polar Construction." MIT Faculty Club, October 30, 8:00 p.m.
Detroit	H. R. Bechwith speaks on "Painting to Sell Your Products." Calvert Hall, November 10, 7:00 p.m.
Pittsburgh	J. A. Hutcheson speaks on "Materials for the Electrical Industry." Westinghouse Research Laboratories, November 11, 8:00 p.m.
Central New York	President Woods speaks on "Polar Construction." Rensselaer Polytechnic Inst., November 17.
Washington, D. C.	President Woods speaks on "Highway Research." November 19, 12:00 m.
Philadelphia	President Woods speaks on "Polar Construction." Villanova College, November 20, 7:00 p.m.



# Technical Committee Notes

## Automotive Rubber

### Master Rubber Specification Being Prepared

A paper on the proposed expanded tabular system for a master rubber specification is being proposed for presentation before the International Rubber Conference in Washington, D. C. in November, 1959 by the ASTM-SAE Joint Committee on Automotive Rubber. The proposal combines existing SAE 10R and ASTM Specifications D 735 tables into a single specification which will consist of two tables. One is made up of all current basic tables, the other all current suffix tables.

The new system, which is being coordinated with the Defense Department's rubber specification program, offers a more flexible way to incorporate new polymers and polymer blends.

A closed-cell vinyl sponge specification is being considered. Upon completion of a gear lubricants study, transmission fluids will be tested for compatibility with certain automotive rubber compounds. The Committee is re-evaluating its method of testing adhesion of vulcanized rubber to metal. A paper on ozone testing is being prepared by Maurice Lowman, Goodyear Tire and Rubber Co., for presentation at the International Rubber Conference in November, 1959. A hypalon table is being developed by a group under the leadership of J. P. Munn, E. I. du Pont de Nemours and Co. A new fluoro elastomers table is being developed by a group headed by D. S. Messinger, Garlock Packing Co.

## Chemical-Resistant Mortars

A means of determining the chemical resistance of mortars has been the primary objective of Committee C-3 on Chemical-Resistant Mortars. At the fall meeting of the committee, held at Watertown, N. Y. on September 11 and 12, a final draft for a proposed method of test was approved subject to committee letter ballot. This accomplishment represents a long period of study and research in establishing a reliable method which can be used by industry. The method covers the measurement of the weight change and change in com-

pressive strength of various chemical-resistant mortars after immersion in chemical solutions and solvents. The results obtained are intended to serve as a guide, but not the sole basis, for the selection of a mortar for a particular application.

Reports were presented indicating work being done on other test procedures related to chemical resistance. A draft of a method of test for flexural strength of silicate mortars has been completed. However, the size of sample involved will require further review. Work is continuing on procedures for determining thermal expansion and shrinkage of chemical-resistant mortars. With the acceptance of the proposed chemical resistance method, it is expected that the development of specifications for each type of chemical-resistant mortar will now proceed with greater acceleration.

Revisions in the group of recommended practices which were published in 1957 were reported. The addition of a paragraph explaining chemical resistance in terms of the particular type of mortar was approved. In the case of the Recommended Practice for Use of Resin-Type Chemical-Resistant Mortars (C 399 T), a table is provided classifying the four general types of resins, namely, furan, phenolic, epoxy, and polyester, as to type of service.

## Standards on Floor Absorbents

THE oil absorptive characteristics of floor absorbents will be studied and research data collected in a cooperative testing program recently initiated by ASTM Committee C-23 on Sorptive Mineral Materials. The program will involve running five replicate determinations on three coded floor absorbents using five recognized test procedures. From this study it is expected that an ASTM standard method will be developed based on the information collected.

This is only one phase of the standardization program launched by one of the newest of ASTM technical committees. A questionnaire on consumer practices has given valuable information and guidance to the committee in selecting work projects. As a result, projects on the agenda of the committee will include the development of methods of sampling and sieve analysis, and for evaluating such properties as free moisture content, loss on ignition, bulk density, absorptive properties, dust content, resistance to breakdown (oil, water, and mechanical), slipperiness, ease of removal, fire resistance, solubility in water, and abrasiveness.

The development of definitions of terms represents another phase of activity in the committee for which a subcommittee has been organized.

Plans have been made to hold a full meeting of the committee on October 16 and 17 in Detroit. Persons or organizations interested in the work of the committee may write to: R. L. Shirley, The Eagle-Picher Co., Insulation Division, 900 American Building, Cincinnati 1, Ohio.

### Papers to Appear in Future Issues of the ASTM BULLETIN

- A Toric Bending Specimen for Investigation of Geometrical Factors in Fatigue—J. A. Bennett and J. G. Weinberg, National Bureau of Standards
- The Fatigue Strength of Magnesium Alloy HK31 as Modified by a Weld Joint—J. E. Breen and A. S. Dwyer, Raytheon Manufacturing Co.
- A High-Temperature, Vacuum, and Controlled-Environment Fatigue Tester—G. J. Danek and M. R. Achter, U. S. Naval Research Laboratory
- Fixtures for Testing Pin-Ended Columns—A. W. Huber, Gruen and Bilfinger, Buenos Aires
- A Note on the Pressurization on the Fatigue Life of Metals—L. W. Hu, The Pennsylvania State University
- False Negative Permanent Strains Observed with Resistance Wire Strain Gages—C. J. Newton, National Bureau of Standards
- Measurement of Bulk Modulus of Hydraulic Fluids—R. L. Peeler and J. Green, California Research Corp.
- Oxidation of Gear Lubes—Laboratory and Field—M. J. Pohorilla and W. Hart, Kendall Refining Co.
- A Method of Test for Potential Efflorescence of Masonry Mortar—P. L. Rogers, Riverton Lime and Stone Co.
- Testing of Prestressing Materials and Concrete Control on the Northern Illinois Toll Highway—J. J. Waddell, Joseph K. Knoerle & Associates, Inc.

# Random Samples...

FROM THE CURRENT MATERIALS NEWS

*From the broad stream of current materials information flowing from "in-box" to "out-box" in a busy editorial office, random samples (mostly random) have been plucked. Thinking them worth re-showing to ASTM'ers who may have missed the original articles, we have included them here. Of course, we had to trim the samples to fit. There will be those who are not satisfied with samples, especially ones which are not really random. But these ASTM'ers can contact the institution, magazine, governmental agency, etc., who placed the original information in the stream, or address Random Samples, ASTM, 1916 Race St., Philadelphia 3, Pa.*

## Hardboard from Sugar Cane Fibers

A \$3,600,000 building board plant soon to be opened in Cuba may point the way to by-product utilization of sugar cane fibers. The process developed by Tayler Corp., a New York engineering firm, is said to make a commercially practicable hardboard from de-pithed bagasse (the cane fiber).

The process is said to be the first to utilize bagasse fiber in making hardboard having the strength of wood. Tests show, according to Tayler, that the bagasse hardboard actually is 15 to 45 per cent stronger than board made from wood pulp. Formerly, hardboard made from bagasse had a relatively low breaking strength and a high degree of water absorption, which confined its use principally to soft insulating board.

The new process overcomes factors heretofore limiting commercially successful production of high-strength hardboard from bagasse by making use of one of the world's most powerful high-frequency dielectric heaters which heats and dries the wet mats of the sugar cane fiber electronically. Mats of depithed pulp will be fed into the unit where a uniform heat is generated throughout the material, raising the temperature to an exact predetermined level and removing all residual moisture. Similar systems are in use in Finland, Sweden, and Israel.

The heated mats go from the unit to a hydraulic press that can exert force of as high as 4000 tons on a board 4 ft by 9 ft. After pressing, the boards are passed through a continuous humidifier which stabilizes them to the normal moisture content of the air.

The process makes possible a wide variety of end products. In addition to hardboard, much of which is expected to go into the making of forms for concrete construction, the factory in Cuba will be able to turn out soft board for insulation, a medium-density board for special furniture requirements, and processed hardboard for simulated tile counters and other decorative and structural uses.

While the product of the Cuban plant is expected to be commercially com-

petitive with wood hardboard manufactured in the United States, the immediate interest is in the Cuban market and the surrounding Caribbean area, where lumber is scarce and expensive and hardboard is almost unknown.

## Synthelastic

NATURAL rubber has now been duplicated in the laboratory. Through the use of newly developed catalysts, chemists are now able to synthesize a substance with structure and characteristics identical to natural rubber. Field tests have shown that the man-made rubber can perform satisfactorily in place of the tree-made product.

Natural rubber is a special form of polyisoprene, a polymer made up of long chains of molecules of the hydrocarbon isoprene. The so-called synthetic rubbers, such as SBR (styrene-butadiene rubber) developed during the war, differ from natural rubber both in chemical structure and in properties. SBR (formerly called GR-S for Government rubber-styrene) wears well for most purposes and accounts for 75 to 80 per cent of all rubber used in passenger car tires. But synthetic rubbers are not suitable for use where thick sections under heavy loads are subjected to rapid and continued flexing that generates considerable heat. Consequently, natural rubber is preferred in such applications as truck and aircraft tires, V-belts, and certain kinds of motor mounts; some types of rubber foam and surgical goods also require natural rubber because of its high strength without reinforcing pigments.

Natural rubber, known as a curiosity for a long time, was not a really useful commodity until Goodyear invented vulcanization (heating together with sulfur) in 1839. Demand soon began to grow rapidly, and there were many attempts to produce a satisfactory synthetic. As early as 1879 the Frenchman Bouchardat obtained isoprene from heating natural rubber and converted it into a rubber-like material; in 1910 Lebedev, in

Russia, produced a rubber-like compound from butadiene. During World War I, the Germans produced so-called methyl rubber (from dimethylbutadiene) by a tedious process, and in this country the first synthetic rubber not made from a hydrocarbon was put on the market in 1929. This was Thiokol rubber, a specialty material obtained through heating ethylene dichloride together with potassium sulfide; today it claims more interest as a component for solid fuels for rockets than as rubber.

The main lines of rubber research, however, continued to emphasize use of the long-chain hydrocarbon building blocks. Sodium and potassium butadiene rubbers (called Buna rubbers) were manufactured in Germany before 1930; production of Buna S, an all purpose rubber prepared from both butadiene and styrene, reached almost 110,000 tons in Germany in 1943. Then the U.S. Government built up its own wartime plants, and by 1945 production of the butadiene-styrene GR-S had passed 790,000 long tons. By 1948, a method of producing "cold" GR-S, at only 41 F, was worked out; combined with fine carbon black, cold GR-S results in tires that have 10 per cent better resistance to abrasion than natural rubber. There are a number of other synthetic rubbers for special purposes—butyl rubber for tire tubes and treads; silicone rubbers for resistance to temperature extremes; and the polyurethane rubbers now used largely in foamed products.

*The rubber market.*—Since World War II natural and synthetic rubber have each taken over about a third of the total rubber market on the basis of their specific properties. In the remainder of the market, either is used, the choice depending on relative prices. In the United States last year, natural rubber accounted for about 37 per cent of the 1.5 million long tons of all types of rubber consumed. The cost of polyisoprene is still too high for it to compete with natural rubber, but markets may grow when low-cost processes for making the isoprene monomer are

developed. Most economists concerned with the problem believe that world demand for natural rubber will increase more rapidly than production, leading to shortages within the next five to ten years. Political events in the Far East could also affect rubber supply; Malaya and Indonesia are the source of approximately 75 per cent of the world's natural rubber.

A major stimulus to research in synthetic natural rubber has been the highly variable price of the natural product. A hundred years ago the average price was 62 cents per lb; in 1910, because of the new demand for automobile tires, it went up to \$3.10 per lb; in 1932, the price fell to 2.6 cents per lb, and now it is around 28 cents per lb.

Although years have been spent in trying to synthesize natural rubber, success has only been recent. Part of the problem stems from the fact that there are eight possible "pure" forms of polyisoprene (not including random mixtures of them), differing in the structure of their molecules and the manner in which they are joined. To produce the desired form—so-called *cis*-1,4-polyisoprene—the isoprene molecules must be linked end to end, or at what are called the 1 and 4 positions, and they must have a particular structure. Ascertaining the structure of the synthesized polymer is difficult; chemists must combine several methods of analysis—infrared absorption, phase microscopy, X-ray diffraction, and chemical.

At present, the only commercial source of the isoprene monomer is the limited quantity obtained as a by-product of oil refining. At least three U.S. companies are making synthetic natural rubber in pilot plant amounts; the processes employed all involve catalysts, but which of the various methods is the best has yet to be decided.

*Industrial Bulletin*, Arthur D. Little, Inc., March, 1958.

### Materials for Semiconductors

SEMICONDUCTORS, including transistors and their near relatives, diodes, are fast growing in importance to the electronics industry. Recent developments in the techniques of purifying germanium and silicon—the most frequently used semiconductor materials—have led to greater reliability and lower cost. Semiconductors are so named because they neither conduct like copper nor insulate like sulfur; their ability to conduct electric current is somewhere in the middle.

Production of diodes and transistors

has increased rapidly in the past few years, from an estimated 14.5 million units in 1954 to about 84.4 million units in 1957; a tenfold increase is anticipated in the next decade. Much of this growth will be accounted for by transistors. In comparison with the vacuum tubes which they are replacing, transistors have the advantages of very small current input requirements, high permissible ambient temperatures, low heat output, extremely long life, excellent durability and very small size. Consumer appliances such as television, radio, and hearing aids used about 19 million transistors in 1957 while industrial and military demands accounted for over 9 million units.

Materials for semiconductors must be very highly purified—impurities may amount to but a few parts per billion. Reliability of a semiconductor depends partly on its purity and partly upon an orderly arrangement of its atoms, which can only be obtained in large single crystals. Consequently, in order to control quality and obtain suitable electrical characteristics, the electronic component manufacturer usually makes his own single crystals, starting from purchased germanium oxide or polycrystalline silicon metal.

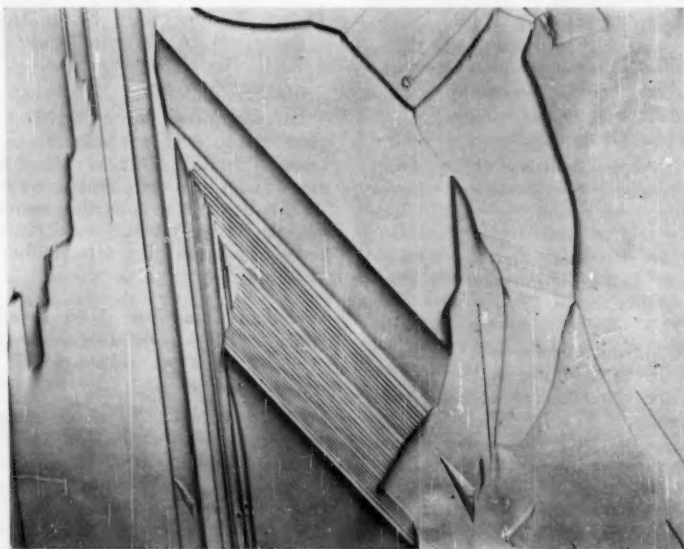
Refining semiconductive materials is almost a laboratory-scale batch process, using a number of very critical processing methods. Moreover, the technology of the field is changing so rapidly that neither a fully standardized product nor full automation has been

achieved. Silicon, very active chemically at high temperatures, melts at 2610 F, which is 850 F higher than the melting point of germanium. Silicon is therefore much more difficult to process. Molten germanium can be held in graphite crucibles, but silicon reacts with the carbon to form silicon carbide. The most suitable material thus far for holding molten silicon is natural quartz, which is much harder to fashion into crucibles. All these difficulties mean high cost; semiconductor-grade germanium costs \$200 to \$240 per lb and silicon averages \$250 per lb.

**Semiconductor supply.**—Despite problems of refining, silicon may become the most widely used semiconductor material. Germanium is thought to be relatively scarce, whereas silicon is the second most plentiful element on earth (oxygen is the first). Germanium is recovered commercially from zinc ores and coal ash. It is found in the United States principally in Illinois and Kentucky; more important sources are located in the Belgian Congo and Southwest Africa.

U. S. refining capacity for germanium is estimated at 85,000 lb per yr; capacity for semiconductor-grade silicon, however, will reach over 150,000 lb by the beginning of 1959. But if the production of transistors increases as expected, even a much greater supply of semiconductor materials will be needed.

*Industrial Bulletin*, Arthur D. Little, Inc., March, 1958.



Twinning in Zone Purified Silicon Rod

Third Prize, Photomicrographs, Black and White—Eleventh ASTM Photographic Exhibit. John H. Miller, Sylvania Electric Products, Inc., Towanda, Pa.



## Petroleum Products and Lubricants

Year Ending June 1958

**T**HERE has been no slackening of the pace of work that is being pursued in ASTM Committee D-2 on Petroleum Products and Lubricants. The period from June, 1957 to June, 1958 found nearly all of the 29 subcommittees active. The 1958 report of the committee to the Society was again the largest report presented. Important information has been published in the new *Special Technical Publications 224*, Chemical Composition of Petroleum Oils; *STP 225*, Knock Characteristics of Pure Hydrocarbons; *STP 214*, Railroad Materials and Lubricating Oils; *STP 221*, Steam Turbine Oils; and *STP 202*, Vapor Phase Oxidation of Gasoline. In 1958, 7 new Tentative Methods and 4 new Standard Methods were approved; 17 Tentatives and 7 Standards were revised, and 13 proposed methods were published as information.

The following sections on Fuels, Lubricants, Other Products, Measurement and Sampling, and Analytical Methods, contain summaries of some of the activities of the committee and of work planned.

### Fuels

**Technical Committee A on Gasoline**, H. M. Smith, chairman; J. B. Rather, Jr., vice-chairman; C. C. Shepherd, secretary

Four subjects continue on the current work program: Specifications for motor gasoline, expression of anti-knock quality above 100 octane number, vapor pressure tests, and the development of better information on gasoline stability.

ASTM specifications for gasoline, D 439, were again reviewed. Increases in the research octane number of regular and premium gasolines were proposed.

Review of CRC work aimed at developing better vapor lock tests continues. So far, no information has been developed that has resulted in a test for correlating fuel properties with vapor lock in equipment that is significantly better than the widely used ASTM Standard Method D 323 for Reid vapor pressure.

Separate methods for obtaining Research and Motor octane numbers above 100 and a reference fuel scale will appear in the 1958 compilation of "ASTM Standards on Petroleum Products and Lubricants."

ASTM Method D 381, existent gum in gasoline, has been revised in order to require heptane washing of all gum residues from motor gasolines.

### Technical Committee E on Burner Fuels

This group is being reorganized under the chairmanship of R. C. Wright of Iron Fireman Co. D. H. Bottrill, Oil Heat Institute of America, Inc., is the new secretary.

Four subcommittees are being considered: Light Fuel Oils, Heavy Fuel Oils, Combustion Tests, and Tests for Pumpability.

On June 25, 1958, a Symposium on Stability of Distillate Fuel Oils was held in Boston, Mass., under the leadership of Wm. deB. Bertolette. Nine papers were given which will shortly be published as a Special Technical Publication.

**Technical Committee F on Diesel Fuels**, W. K. Simpson, chairman; V. C. Barth, vice-chairman; Earle Freeman, secretary

The committee has continued a review of the ASTM Classification for Diesel Fuel Oils, D 975. Surveys of diesel fuel oils in the market have been studied to find out how they compare with the type of fuels in the classification looking toward the formulation of revisions.

**Technical Committee J on Aviation Fuels**, J. J. Harrington, chairman; R. D. Kelly, vice-chairman; T. B. Rendel, secretary

Although, in 1957, minor changes were made in the specifications for aviation gasoline, D 910, the main interest and emphasis has been in work on aviation turbine fuels. In 1957, a proposed specification was published as information that outlines two possible commercial turbine fuels. Comments on this proposed specification resulted in changes in the ver-

sion to be republished in 1958. Discussion centers on the specification of a kerosine-type fuel of lower freezing point than the -40 F already suggested. Other items in the proposed specification also are being reviewed.

Concurrent with the talks on turbine fuel specifications, Technical Committee J is developing a series of ASTM test methods that may help to better define turbine fuel quality. Included are methods for flow properties at low temperatures (pumpability), particulate matter, filterability, smoke point, fuel composition by hydrocarbon type, and thermal stability. Of these, the smoke point method has been standardized (ASTM D 1322) and tests for thermal stability by CRC coker, particulate matter, and filterability published as information (1958).

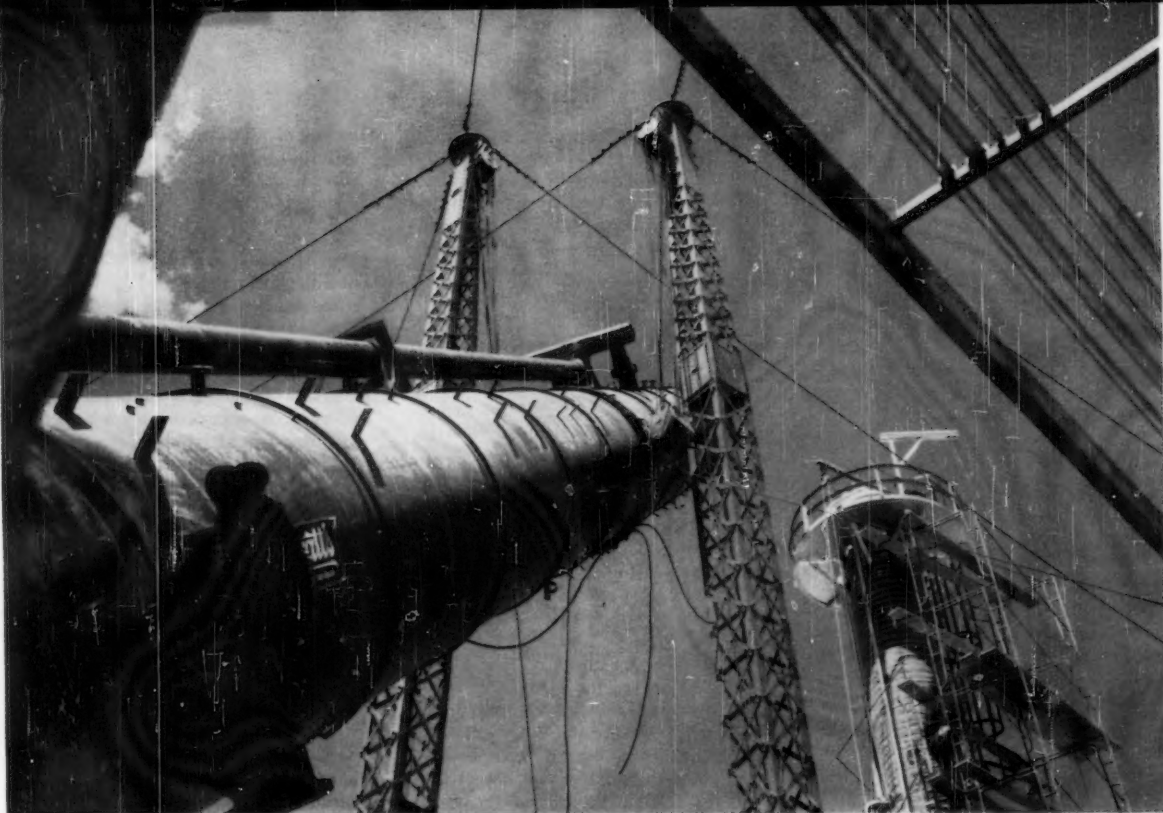
**Research Division I on Combustion Characteristics**, H. M. Trimble, chairman; R. L. Kirkpatrick, vice-chairman; Josiah French, secretary

The programs of the Division are directed toward the improvement of the existing ASTM engine test methods for relating fuels and the investigation of new equipment and instruments for this purpose.

New piston rings have been approved for the aviation and supercharge methods and are being recommended for the motor and research methods. The new ring assembly features a chromium-plated top ring and two narrow-faced oil control rings which are said to reduce wear. ASTM-CFR engines may now be used in ASTM Methods D 357, D 908, D 613, D 614, and D 909 with cylinders of up to 0.030 in. oversize.

Although procedures for determining octane numbers above 100 by motor and research methods are not ready for full ASTM standardization, details of the scale above 100, the reference fuel system, and the motor and research methods for rating fuels above 100 octane number have been published as information.

Improved new equipment and instrumentation for use in determining octane numbers above 100 are receiving considerable attention. In addition, ways and means of bettering present



Going Up (Erecting Refinery Fraction Column)

Third Prize, General, Black and White—Eleventh ASTM Photographic Exhibit. Edmund F. Hawes, Universal Oil Products Co., Des Plaines, Ill.

equipment and procedures are not being overlooked. The Division is cooperating with Technical Committee A on Gasoline in developing a panel discussion on the art of knock testing at higher octane number levels to be held in Washington, D. C., on October 7, 1958.

A few of the items that are the subject of study are: different cylinder configurations, more precisely blended reference fuels, instruments to measure various combustion phenomena, and the relationship between fuel and the engine in which it operates. The work of the Coordinating Research Council, Inc., which involves a study of several combustion phenomena is being constantly reviewed through close contact.

#### Research Division V on Analysis of Fuels, E. T. Scafe, chairman

This Division is active on a program to improve the ASTM methods for determining deterioration products and extraneous materials in fuels, and to develop new standards. Close cooperation with Technical Committee A on Gasoline, F on Diesel Fuels, and J on Aviation Fuels is maintained.

The test for existent gum in gasoline, D 381, was revised. Method D 95, water in petroleum products, was made a full standard.

Two new methods, (1) filterability of aviation turbine fuels, and (2) particulate matter in hydrocarbons, were developed at the request of Technical Committee J.

A cooperative program that involves four residual fuel blends is under way in order to investigate Method D 473, sediment by extraction. Evaluation of the results may suggest improvements in this standard.

Study of an electron microscope method for evaluation of diesel fuels continues. Users and producers of diesel fuels are participating in the development of the method, and Technical Committee F on Diesel Fuels is being informed of the progress of the work. Variables in specimen preparation and in the rating system for fuel samples are receiving attention.

#### Research Division VIII on Volatility, R. J. Askevold, chairman; G. W. Jack, secretary

Discussion and study of improvements in the Distillation Methods D 86, D 158, and D 216 continue to be an important part of the current activity. The proposed combined distillation method applicable to products now covered by Methods D 86 and D 158 was republished for information in 1958, with revisions. The procedure for reduced-pressure distillation, D 1160, was

placed under Section A on Distillation and its original sponsor, Section E on Vacuum Distillation has been discharged.

With the publication of a completely revised Pensky-Martens closed cup flash point test (D 93) in 1958, the Division completed the modernization of the flash point methods under the jurisdiction of Committee D-2. The other two methods D 56, tag closed cup flash test, and D 92, Cleveland open cup flash test, had been revised in 1956 and 1957. Further improvements in these tests will be discussed.

Improvements were made in the Reid vapor pressure test, D 323, particularly in the section on precision. Other changes in D 323 and in the test for vapor pressure of liquefied petroleum gases, D 1267, are being considered. These include vapor pressure - temperature corrections for D 1267, and consideration of micro apparatus and procedures.

#### Research Division X on Corrosion Tests, F. D. Tuemmler, chairman

Arrangements were completed for the replenishment of the stock of ASTM Copper Corrosion Standards. These comparison standards have proved to be helpful in the interpretation of results of the standard copper corrosion test, Method D 130.

The group studying humidity cabinet tests is continuing cooperative work that is looking into variables believed responsible for unacceptable precision. The rate of condensation of water on test panels and those factors affecting it are being reviewed.

## Lubricants

**Technical Committee B on Lubricating Oils**, *W. S. James, chairman; W. A. Wright, secretary*

Some important work was accomplished in 1957-1958, and the program for next year calls for increased activity. The ASTM tests for foaming characteristics of crankcase oils, D 892, and for dilution of crankcase oils, D 322, were revised. The foam test now includes procedures for determining pore size and air permeability of the diffuser stones. Method D 322 was improved by better specification of temperature prior to testing and of the cooling period. The program on crankcase dilution tests also included the publication as information of a new vacuum distillation method.

An important development centers around the presentation to Technical Committee B of a report compiled by automotive representatives on Section G-IV, Application of Crankcase Oils. The report contains a description of engine operating conditions currently representative of service described by service classification MS, as set forth in the Lubricant Service Classification published by the American Petroleum Inst. Five engine test sequences for describing Service MS have been developed. Complete review and discussion of the information is taking place in Technical Committee B and in other interested groups.

There has been considerable work done on an investigation of scales for expressing the viscosity-temperature relationships of petroleum oils. Present direction of these studies points to (1) a revision of the present method, D 567, and charts, D 341, for expressing viscosity index to eliminate faults in the region above 100 V.I., and (2) the development of a more fundamental system for those whose needs are not met by a relative system.

The work of Section G-IV on Application of Crankcase Oils will be expanded to include study of the equipment requirements for transmission and rear-axle lubricants.

**Technical Committee C on Turbine Oils**, *G. V. Browning, chairman; C. M. Loane, secretary*

Six sections and a study group have been engaged in a program covering de-

sign of turbine lubricating systems, a joint activity with the ASME, and the development of better oxidation tests, emulsion tests, and rust tests for turbine oils. Also, load-carrying capacity, compatibility, and fire resistance are being investigated.

Recommendations have been prepared on the design of gravity, pressure, and gravity-pressure lubricating systems for turbines.

The standard rust test, D 665, may be revised to make it applicable to synthetic turbine fluids that are heavier than water.

The short-term oxidation test, published as information in 1956, does not appear to satisfy all groups represented in Technical Committee C. The test was developed to supplement the standard method D 943, oxidation of inhibited steam-turbine oils. Reasons for dissatisfaction will be investigated.

Method D 1401, emulsion characteristics of steam turbine oils and other emulsion tests are being compared and evaluated.

Technical Committee C will continue its study of interest in and use of fire-resistant turbine fluids and the problems attendant with their application.

**Technical Committee G on Lubricating Grease**, *C. L. Pope, chairman; E. W. Adams, first vice-chairman; H. T. Peebles, second vice-chairman; P. R. McCarthy, secretary*

Method D 1092, apparent viscosity of lubricating grease, was revised by widening the temperature range and including a new statement of precision. Method D 1402, effect of copper on the oxidation rate of grease, was adopted as ASTM Standard. Information on analysis of greases for which the procedures outlined in Methods D 128 are unsuitable were republished as an Appendix to D 128.

Considerable work is being done on methods for cone penetration, and results of cooperative programs are being evaluated. A test to determine the tendency of grease to separate oil when stored in containers is being investigated. A report "Oil Separation from Lubricating Greases in Storage" is being published in the BULLETIN. A new group will study the development of a test method for shear stability of greases by the roll test procedure.

Cooperative tests have been run on an improved high-temperature anti-friction bearing grease tester, but the results indicate that other variables still need study before a precise test can be written.

Work has been started on a test to measure the rust-prevention properties of lubricating greases.

**Technical Committee K on Cutting Fluids**, *L. B. Sargent, Jr., chairman; E. A. Reehl, vice-chairman; R. G. Moyer, secretary*

A program to develop chemical, physical, and field tests for cutting fluids is continuing.

The method for active sulfur in cutting fluids, published as information in 1956, has been reviewed and will be proposed as an ASTM tentative. Methods for active chlorine are still being studied and new cooperative work is planned.

Flank wear and finish tests are also continuing to receive attention. Cooperative programs are in progress, the results of which will be ready for review early in 1959.

A program of testing centered on the development of the Falex film-strength test is being continued. A revised procedure which shows promise of better precision is being studied.

A cooperative program on rusting tests for soluble cutting fluids has been planned in order to develop a rusting test applicable to cutting fluids.

**Technical Committee N on Hydraulic Fluids**, *D. B. Gardiner, chairman; L. W. Manley, vice-chairman; W. H. Millett, secretary*

Eight active sections have been at work during the year. Seven separate cooperative programs are in progress to develop information on various characteristics of nine reference hydraulic fluids by mutually accepted procedures.

The nine fluids include a petroleum-base oil, five fire-resistant fluids of varying type, and three synthetic fluids not classified as "fire-resistant." Information is being obtained on the measurement of flammability characteristics, lubricity, oxidation and thermal stability, hydrolytic stability, and compatibility (including compatibility with rubber and protective coatings).

Three informal symposiums on aircraft applications of hydraulic fluids have been held at which information on bulk modulus, contamination, decomposition, filtering, and hydraulic testing, has been presented and discussed. An informal symposium on industrial applications of hydraulic fluids is planned in 1959, and the whole committee will take part in the October, 1959, Third Pacific Area National meeting of the Society in San Francisco, Calif., by preparing a formal ASTM Symposium on Hydraulic Fluids.

Other work has included a survey of existing ASTM methods applicable to the testing of hydraulic fluids, a study of causes and origin of fires believed caused by hydraulic fluids, and an intensive program on flammability test methods.



**Research Division VI on Analysis of Lubricants, J. B. Rather, Jr., chairman**

In 1958, several revisions were made in the ASTM color-indicator method for neutralization value, D 974. Benzene was replaced by the less toxic toluene both in D 974 and in the companion method, D 664. Specifications for the color-indicator, *p*-naphtholbenzein, were included; the precision section was made applicable only to light-colored oils.

A cooperative program to establish the precision of Method D 91, precipitation number of lubricating oils, was completed. Also, the method was compared with an alternative procedure that specifies new pear-shaped centrifuge tubes with more easily-read graduated tips, and the use of pure *n*-heptane as the solvent.

Carbon residue Methods D 524, Ramsbottom, and D 189, Conradson, were revised in order to permit the testing of samples with carbon residues as high as 20 per cent.

Future work includes a discussion and decision of whether or not to reinstate the now withdrawn procedure for neutralization number using phenolphthalein as indicator (D 663), a decision to either revise Method D 91 or publish a new procedure for insolubles in lubricating oil, and the expansion of the correlation between the Ramsbottom and Conradson carbon residue tests.

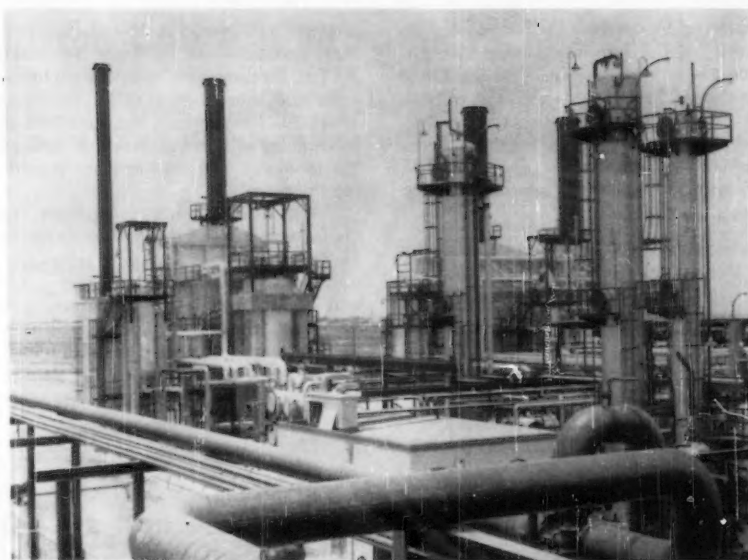
**Research Division VII on Flow Properties, J. C. Geniesse, chairman**

The division continued to cooperate with Technical Committee B in the development of improved methods for expressing the viscosity-temperature relationships of petroleum oils. Method D 666, conversion of kinematic viscosity to Saybolt furol viscosity was extended to provide conversions at 210 F.

The group recommended the establishment of a series of ASTM oil standards for viscometer calibration to replace the American Petroleum Inst. oil standards now referred to in the kinematic (D 445) and Saybolt (D 88) methods. The recommendation is being considered by the ASTM. The API has expressed a willingness to discontinue its viscosity standards if suitable arrangements for ASTM oil standards can be made.

**Research Division IX on Color, H. M. Hancock, chairman**

Color methods D 155 (ASTM-Union), D 156 (Saybolt) and the new Method D 1500 were reviewed. Editorial changes were made in the two older methods. The new method was revised in order to permit wider tolerances for the ASTM color glasses.



**Catalytic Reformer Unit**

*Eleventh ASTM Photographic Exhibit. John H. Rudd, Richfield Oil Corp., Wilmington, Calif.*

## Other Products

Important petroleum products such as light hydrocarbons, wax, sulfonates, and petrolatum are subjected to many tests. The ASTM has developed many of these, the maintenance of which is the responsibility of such Committee D-2 groups as Technical Committee M on Wax, Technical Committee H on Light Hydrocarbons, and others.

**Technical Committee H on Light Hydrocarbons, C. W. Ricker, Jr., chairman; R. C. Alden, Secretary**

The committee completed the ASTM Butadiene Measurement Tables, D 1550. Although test methods for butadiene represent the results of past standardization activities, an ambitious program to develop methods of test for other light hydrocarbons such as ethylene, isobutylene, isoprene, and cracked butanes is now in progress.

Test methods for liquefied petroleum gas such as specific gravity, corrosion, and weathering continue to receive attention.

The scope of several of the existing methods of test for butadiene will be revised to include concentrates containing over 60 per cent butadiene.

**Technical Committee M on Wax, G. Rumberger, chairman; R. P. Daniels, first vice-chairman; M. W. Kane, second vice-chairman; W. J. Yates, secretary; D. I. Walker, assistant secretary**

Two new methods were developed by the committee, which functions as a

joint activity of the ASTM and the Technical Association of the Pulp and Paper Industry. The methods describe tests for breaking strength and sealing strength of paraffin waxes, and were published as information in the 1958 Report of Committee D-2.

The work of the committee has resulted in the development of several new ASTM-TAPPI methods for physical and functional properties of wax. Accomplishments to date include publication of ASTM-TAPPI Methods for melting point, D 87 and D 127; oil content, D 721; tensile strength, D 1320; needle penetration, D 1321; and blocking point, D 1465. Proposed methods on odor, gloss, and oxidation stability have been published as information. New tests are being considered, including a procedure for measuring the friction between waxed surfaces.

An important Symposium on Wax was held at the February, 1958, Annual Meeting of TAPPI. Papers presented covered the structure and composition of petroleum waxes and their relationship to performance characteristics. The papers appeared in the June, 1958, issue of TAPPI magazine and have been made available by the ASTM as a special reprint.

**Subcommittee I on Pharmaceutical Tests, C. F. W. Gebelein, chairman**

The test for carbonizable substances in paraffin wax, D 612, will be reviewed.

A special liaison group, which had been appointed by the Advisory Com-

mittee of Committee D-2, completed its work. The group was given the assignment of rendering advice and assistance to the 1960 revision committees of both the U. S. Pharmacopeia and the National Formulary concerning the modernization of information about ASTM tests and specifications referred to in the two publications.

**Subcommittee XXIV on Tests for Petroleum Sulfonates, C. F. W. Gebelein, chairman**

Improvements have been suggested in analysis and techniques, and these will be evaluated.

## Measurement and Sampling

**Research Division II on Measurement and Sampling, P. L. DeVerter, chairman; W. E. Scovill, vice-chairman; B. J. Heinrich, vice-chairman; E. F. Wagner, secretary**

The division completed work on new tentative tables for volume correction of benzene, toluene and para-xylene, D 1555, which were prepared jointly with ASTM Committee D-16. Method D 96, water and sediment by centrifuge, was changed to require that benzene saturated with water at test temperature be used. Methods D 1220, calibrating liquid containers, and D 1408, calibration of spherical tanks, were revised to provide information on tank volume changes caused by thermal expansion of tank shells fabricated from materials other than low-carbon steel. A full program at work on tank calibration is continuing.

Tables for reduction of specific gravity and volume to 60 F for liquefied petroleum gases have been prepared. They will be issued as Tables 33 and 34 as an amendment to the American Edition of the ASTM-IP Petroleum Measurement Tables (ASTM D 1250; IP 200).

A Symposium on Bulk Quality Measurement was held in February, 1958. In view of insistent requests for copies of the papers presented, the ASTM may be asked to issue this symposium as a Special Technical Publication.

Study is being made on variations of pipe line displacement. Work is being done on the precision of the test for specific gravity of liquefied petroleum gases using a hydrometer. Efforts are being directed towards the publication of such a test by the ASTM as soon as possible.

In cooperation with Committee E-1 on Methods of Testing, a number of

changes were made in ASTM Hydrometer Specifications in order to make ASTM Hydrometers more in conformance with those used in other countries. Work on temperature measurement is continuing with investigation of various thermometer and temperature recording instruments.

Work on sampling procedures includes a revision of the ASTM standard D 270, method of sampling petroleum and petroleum products. More work will be done on Method D 96, water in petroleum products and other bituminous materials, which will include investigation of centrifuge tubes of new shapes, and permissible use of certain de-emulsifiers and other solvents.

## Analytical

**Research Division III on Elemental Analysis, C. M. Gambrill, chairman; R. O. Clark, vice-chairman; D. C. Roberson, secretary; W. V. Cropper, assistant secretary**

The results of several years of work in Research Division III reached fruition, with the publication of four new tentative methods: vanadium in Navy special fuel oil, D 1548; zinc in new lubricating oils and lubricating oil additives, D 1549; sulfur in petroleum products by rapid high-temperature combustion, D 1552; and sulfur in petroleum products by the quartz tube method, D 1551. A proposed replacement for the standard method for ash content of petroleum oils, D 482, was published as information as an Appendix to the 1958 Report of Committee D-2. Revisions were proposed in Method D 129, test for sulfur in petroleum products and lubricants by the bomb method, and D 1368, tetraethyllead in primary reference fuels.

Work will be done on the determination of trace sulfur concentrations in fuels and liquefied petroleum gases. A high-temperature combustion method for the determination of chlorine will be reviewed. A cooperative program to evaluate a colorimetric procedure for phosphorus is being completed. Compleximetric titration methods for barium, calcium, and zinc in lubricating oils are receiving attention. Quicker methods for tetraethyllead in gasoline, such as flame photometric procedures, will be studied. Work will be done on the development of an ASTM method for water in petroleum products. A review will be made of ways to improve the accuracy of the lamp method for hydrogen, oxygen, and nitrogen, D 1018.

**Research Division IV on Hydrocarbon Analysis, J. F. Hickerson, chairman; E. T. Scafe, vice-chairman; R. A. Klett, secretary; A. J. Ackerman, assistant secretary**

The division developed a method for hydrocarbon types in non-olefinic gasolines by mass spectrometer that was published as information as an Appendix to the 1958 Report of Committee D-2. Method D 1019, test for olefinic plus aromatic hydrocarbons in petroleum distillates, was revised to clarify the position of the sulfonation flask in the shaking machine. The use of dyed gel was permitted in Method D 1319. A new Section M on Mass Spectrometry Methods was established. Work will be done on the precision section of Method D 1019 and the cooperative testing of a proposed revision of Method D 875. A major revision of the electro-metric method, D 1159, is under way which envisions the eventual withdrawal of D 1158, the companion colorimetric method. Further work will be done to improve the precision and utility of the method for hydrocarbon types, D 1319. Cooperative programs for the improvement of the method has an international flavor, since data have been received from other countries. The division will consider a consolidation of several of the density methods for petroleum products that are now published by the Society. Three new mass spectrometry methods are being considered for ASTM standardization. Two are for determination of hydrocarbon types in gasoline (one is for low-olefinic stocks and the other for cracked stocks). The third method, which was published as information in 1957, is for the determination of aromatics by carbon number. Consideration is still being given to a compilation of the hydrocarbon type analysis methods, which will include in one publication all of the methods that have been developed by Committee D-2 for this purpose as well as related supplementary information.

An important new activity has been the increase of work on gas chromatography. Terms and definitions in the field of gas chromatography have been compiled and submitted to *Analytical Chemistry* for publication. The group is working on the standardization of a test for the analysis of butanes by this technique. Gas chromatography procedures are being reviewed and systematized.

Other work includes a study of convenient means of deparaffinization of test samples and investigation on methods for the separation of saturated hydrocarbons from gasoline samples.

# Separation of Oil from Lubricating Greases in Storage

A Report of Section IV on Lubricating Grease Research Techniques<sup>1</sup> of Technical Committee G of ASTM Committee D-2 on Petroleum Products and Lubricants

**O**IL SEPARATION from lubricating greases has long been recognized by manufacturers and consumers as an undesirable feature accompanying lubricating grease storage. Technical Committee G of ASTM Committee D-2 recognized many years ago that a test method was needed to measure the tendency of lubricating greases to separate oil. In December, 1948, Subsection 6 on Storage Stability of Greases was established under Section II on Consistency Measurements and Related Physical Tests for Lubricating Greases, to develop a satisfactory test method.

Subsection 6 studied a temperature cycling procedure (40 to 120 F), but found only slightly more oil separation than in constant-temperature tests. Several laboratory tests were found to give reproducible results, but these did not correlate with data obtained in 35-lb pail storage tests.

Because of these puzzling and contradictory findings, The Advisory Sec-

tion of Technical Committee G on Lubricating Grease in 1952 requested Section IV on Lubricating Grease Research Techniques to investigate the mechanism of oil separation from lubricating greases and to suggest suitable techniques or methods for measuring the tendency of greases in storage to separate oil.

Section IV began work on this problem in 1953 on completion of the investigation of thixotropy in greases. Section IV agreed to study the mechanism of oil separation from lubricating greases and suitable techniques for measuring the tendency of greases in storage to separate oil.

## STUDIES OF MECHANISM OF OIL SEPARATION AND VARIABLES INVOLVED

### Stresses Affect Oil Separation

#### Differential Pressure Accelerates Oil Separation

It has been known for over twenty years that in certain tests (1)<sup>2</sup> an increase of pressure over atmospheric increases oil separation from lubricating greases. Pressure can be applied by means of weights, gas pressure, static head of the oil in the grease, centrifugation, etc.

In an investigation of the pressure variable, 50-g samples of grease were placed in 3-oz. Gill-type ointment tins equipped with 40-mesh screen bottoms. The oil separating from the grease samples, subjected to various loads (by means of brass weights), was measured over a period of time. The results of these experiments are given in Table I and are plotted in Figs. 1 and 2.

The conclusions drawn from this test are as follows:

(a) The rate of oil separation de-

creased as the test proceeded, see Fig. 1.

(b) With the samples containing added weights, there is an approximately linear relationship between added weights and the amount of oil separated in a given time, see Fig. 2.

(c) No oil separation occurred without added weights. Thus, the effective static head in the sample without weights was less than expected. This is thought to be due to "bridging" of the grease structure supported by the container surfaces, as will be discussed further.

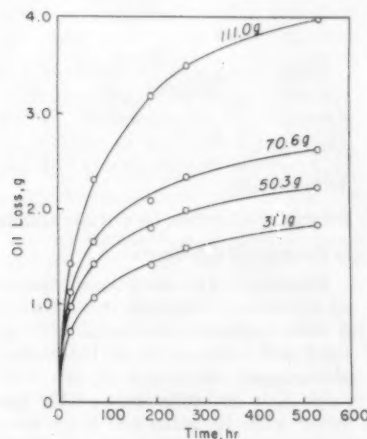


Fig. 1.—Effect of time on oil separation.

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<sup>1</sup> B. B. Farrington, California Research Corp., Chairman; H. A. Woods, Shell Oil Co., Vice-chairman; J. R. Allison, Leffingwell Chemical Co.; G. H. Hommer, Tidewater Oil Co.; B. Y. McCarty, The Texas Company; W. B. Mullin, General Petroleum Corp.; H. J. Worth, Union Oil Co.; H. C. Zweifel, Richfield Oil Corp.

<sup>2</sup> The boldface numbers in parentheses refer to the list of references appended to this paper.

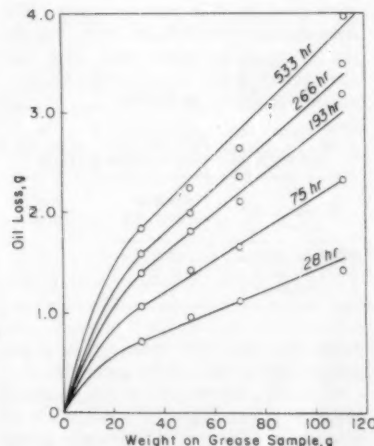


Fig. 2.—Effect of weights on oil separation.

The effect of air pressure on oil separation was shown by the Socony Mobil Pressure Test (7). A 150-g sample of grease was placed in a filter funnel containing a porous plate. When air pressure was applied to the grease, oil seeped through the plate. The data in Table II show that increasing air pressure increased the rate of oil separation, but not in direct proportion. In these experiments, oil separation rate was nearly proportional to the square root of the pressure. This is also true for the weight load experiments.

TABLE II.—EFFECT OF AIR PRESSURE ON OIL SEPARATION RATE.

Air Pressure, psi	Oil Lost in 20 hr, g
0.0	0.0
0.41	1.0
2.5	2.4
4.8	3.2
7.3	4.0

TABLE I.—EFFECT OF WEIGHTS ON OIL SEPARATION.

Sample weight, 50 g					
Sample	No. 1	No. 2	No. 3	No. 4	No. 5
Weight on grease surface, g	0.0	31.1	50.3	70.6	111.0
Grams of oil separated in:					
28.5 hr.	0.0	0.71	0.96	1.12	1.41
75.0 hr.	0.0	1.06	1.42	1.65	2.31
193.0 hr.	0.0	1.40	1.80	2.10	3.19
266.5 hr.	0.0	1.59	1.99	2.35	3.50
533.0 hr.	0.0	1.84	2.24	2.64	3.98



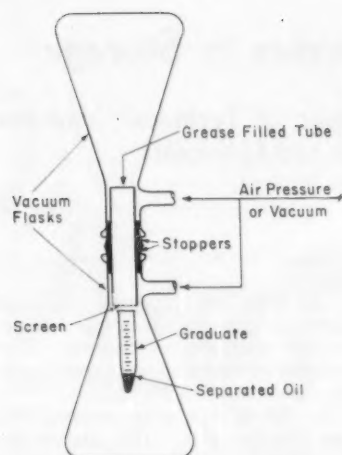


Fig. 3.—Apparatus for measuring effect of differential pressure on oil separation from greases.

In an experiment involving hydrostatic head, cone-shaped depressions were made in 35-lb pails of grease. The cone depths were 2.5 and 5 in., but the diameters of the cones were such that the surface areas were equal. Oil separation is shown in Table III. The oil

TABLE III.—EFFECT OF CRATER DEPTH ON OIL SEPARATION.

Oil Separation, ml	Shallow Crater, ml	Deep Crater, ml
After 30 days...	0.8	4.3
After 60 days...	1.2	10.0
After 90 days...	1.7	15.7

separation rate was increased approximately as the cube of the grease head.

Centrifugal force can act as a source of pressure to cause greatly accelerated oil separation. Very small grease samples centrifuged on a filter showed higher percentages of oil separation than did the same greases in other tests conducted at lower pressures for much longer periods of time.

#### Tension Stops or Reduces Oil Separation

The effect of tension on oil separation from lubricating greases was originally noted by one of the section members who inverted cans of lubricating grease and found that no oil separated. This effect was further investigated by inverting cylinders of grease. Ten greases were studied at  $77 \pm 3$  F and at  $100 \pm 3$  F. These cylinders,  $3\frac{1}{4}$  in. in diameter and  $4\frac{1}{2}$  in. in depth, having closed bottoms were filled with the test greases. No oil separation was observed at either test temperature on any of the greases.

In order to study the effect of differ-

<sup>1</sup> Tentative Method of Test for Cone Penetration of Lubricating Grease (D 217 - 52T), 1955 Book of ASTM Standards, Part 5, p. 127.

ential pressures on the top and bottom surfaces of cylinders of lubricating grease, an apparatus was constructed as shown in Fig. 3. This consisted of a steel cylinder  $\frac{3}{4}$  in. in diameter and 6 in. long, covered at one end with 80-mesh copper wire screen. Two 2000-ml suction flasks were attached to the steel cylinder by two rubber stoppers mounted back-to-back on the central portion of the cylinder. The apparatus was so arranged that, when assembled, the cylinder was vertically supported with the screen at the lower end having the graduated centrifuge tube suspended below it. The pressure differential was restricted to pressures less than 0.5 psi, as a greater differential pressure caused grease to come through the screen with the separated oil. To insure against this possibility, this series of tests was conducted at a pressure differential of 0.25 psi. Reduced pressure could be applied either to the upper or lower suction flask. The duration of the test was 500 hr at  $77 \pm 3$  F. The tendency of oil to separate under various conditions was as follows:

1. No differential pressure between the ends of the cylinder. Normal oil separation.
2. Subatmospheric pressure applied to the lower end with the upper end at atmospheric pressure. Increased oil separation, and
3. Subatmospheric pressure applied to the upper end with the lower end at atmospheric pressure. No oil separation.

Greases were studied as illustrated in Fig. 4. Oil separation was always reduced by tension. Tests illustrated in Fig. 4 (b) separated 10 to 50 per cent as much oil as those in Fig. 4 (a). No separation occurred when greases in tension, as shown in Fig. 4 (c), were tested for three months.

#### Temperature Increase Accelerates Oil Separation

With samples from three types of greases studied by the committee in this program (Table VI), the average ratio of oil separation rates at 77 and 100 F was 1.7 for results obtained in the Cratered Pail Oil Separation Test described later. The data are shown in Table IV.

TABLE IV.—EFFECT OF TEMPERATURE ON OIL SEPARATION RATE

Oil Separation Time, weeks	Ratio of: Oil Separation Rate at 100 F Oil Separation Rate at 77 F		
	Grease TG-IV-10	Grease TG-IV-11	Grease TG-IV-12
1.....	1.9	2.0	1.8
2.....	1.8	1.9	1.6
3.....	1.7	1.7	1.5
4.....	1.6	1.7	1.4

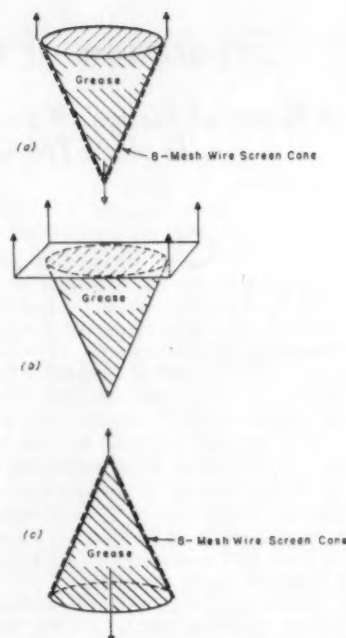


Fig. 4.—Tension reduces or stops oil separation.

- (a) Normal oil separation, grease packed in screen cone
- (b) Tension reduces oil separation, grease hanging from horizontal surface without screen cone
- (c) Tension stops oil separation, grease packed in inverted 8-mesh wire screen cone

Phase changes in the thickener with temperature interrupt the normal oil separation rate-temperature curves. This effect is described in reference (2) for a series of stearate greases with four different cations.

#### Effects of Vibration on Oil Separation

##### In Commercial Containers

Vibration had an accelerating effect on oil separation from grease in 35-lb pails. A grease containing an oil of 270 SSU at 100 F and having a 360 ASTM worked penetration<sup>3</sup> was stored in two 35-lb pails. One pail stood near a 3450 rpm motor, while the other was in the same area but was not subjected to vibration. The pails were equipped with  $\frac{1}{4}$ -in. diameter standpipes extending through the bottom and to a height of 4 in. below the top edges of the pails. The grease was cratered from the edges of the pails to the top of the standpipe.

The rate of oil separation from the vibrated sample was greater for the first 50 days than from the stationary sample; the reverse was true for the remaining 45 days. At the end of the 95-day test, 128.7 g of oil had separated from the vibrated sample, compared to 114.8 g from the stationary sample.

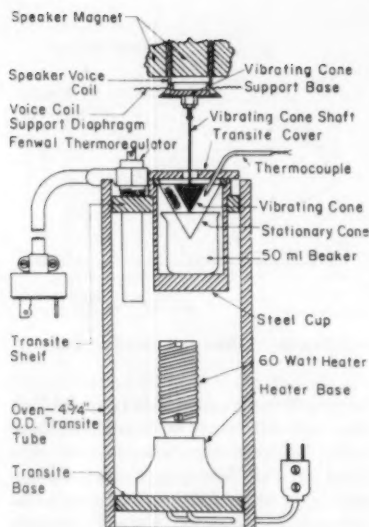


Fig. 5.—Grease vibration apparatus.

#### Laboratory Studies, Effects of Frequency and Power Input

As greases are often subjected to vibration during storage, an apparatus was constructed to study the effect of vibration at various frequencies and power-input levels on the tendency of grease to separate oil. A sketch of the vibrating and stationary components of the apparatus is presented in Fig. 5. Briefly, this instrument consisted of a vibrating cone placed concentrically within a stationary cone, both being within an oven which was heated under thermostatic control. The stationary cone was filled with grease and the top assembly raised to embed the vibrating cone into the grease until its top edge was flush with the top of the grease. The two cones were then concentrically positioned. The oven was heated to 125 F, the inner cone vibrated for 8 hr at a given power input and frequency, and the amount of oil separation measured. Typical test results are presented in Table V.

At constant power-input levels, oil separation tended to be lower at the lower frequency, where amplitudes of vibration were greater.

TABLE V.—EFFECT OF VIBRATION ON OIL SEPARATION—CONSTANT FREQUENCY, VARIABLE POWER-INPUTS.

National Lubricating Grease Institute (NLGI) No. 2 Grade Lithium Soap Grease.

Frequency, cycles per sec	Power-Input, milliwatts	Oil Separated at 125 F, g
0	0	0.54
60	3.2	0.51
60	32	0.22
60	100	0.11
60	320	0.12
180	3.2	0.50
180	10	0.42
180	32	0.40
180	100	0.18

#### Migration Pattern During Oil Separation

In order to gain some insight into the mechanism of oil separation from lubricating greases, experiments were set up to determine if oil separated only from the area adjacent to the surface or if there was a general migration throughout the grease. These studies were made with oil-soluble dyes and radioactive tracers.

#### Oil-Soluble Dyes

A transparent plastic cell (see Fig. 6) was filled with grease, a portion of which was dyed with an oil-soluble dye and charged into the center of the cell as a strip bordered on both sides by the undyed portion. The cell was tipped to an angle of 45 deg and held in that position in an oven at 100 F for several weeks.

The dyed portions above the plane of flow showed a slight general shifting downward. The portion below the horizontal plane showed no movement. Neither did the dyed strip in the similar cell which was stored in an upright position with the open end on top.

From these tests it is concluded that oil separation is the result of the force of gravity which causes a flow of the oil throughout the affected mass of grease.

#### Radioactive Tracers

Internal migration of oil was explored by injecting radioactive antimony (triphenylstibene) with a hypodermic syringe into selected regions of a grease sample. An attempt was made to study fluid migration by tracing the movement of the radioactivity. But cracking of the grease took place during the experiment and the data obtained were not precise enough to permit conclusions to be reached concerning oil migration.

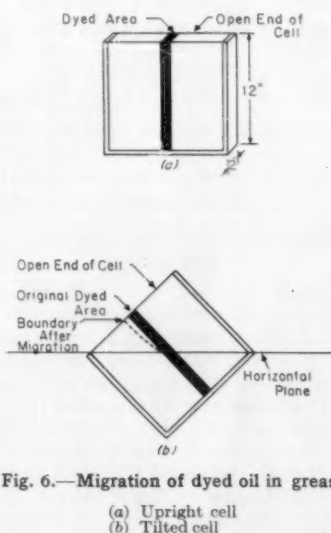


Fig. 6.—Migration of dyed oil in grease.

(a) Upright cell  
(b) Tilted cell

#### TESTS FOR MEASURING OIL SEPARATION DURING STORAGE

One of the principal objectives of the committee was to establish primary standards of oil separation in commercial-sized packages. Previous work by Subsection 6 showed the need for a reproducible storage stability test which would not require too large a sample of grease and yet represent commercial-sized packages. For this reason, several types of 35-lb pail storage tests were considered, two of which are described in detail below. In addition, a correlation was needed between a primary storage stability test and an accelerated laboratory bench test. Although several laboratory tests for oil separation had been previously studied, the unreproducibility of the pail test at that time prevented good correlation.

#### Primary Standard Test for Oil Separation During Storage

As in the previous Subsection 6 work, 35-lb pails were chosen for primary storage stability tests because they represent commercial practice, yet are small enough for laboratory experiments. Two types of pail tests were conducted, and these are described under the appropriate headings below.

#### Crater Test

Early experiments by Section IV with crater-type tests used either (1) single, shallow craters with a drain pipe through the grease and the bottom of the pail, or (2) a single crater with apex at a hole in the bottom of the pail through which oil dripped.

Several variables affecting oil separation in crater-type tests were studied and the following conclusions reached:

- (a) hydrostatic head was a major factor in oil separation (see Table III).
- (b) Oil removal from craters during the test period caused erratic results and increased bleeding. Thus, it was necessary to measure the amount of separated oils by "out gage" system, and
- (c) Screens were found necessary to keep soft greases from slumping into the craters.

A crater-type oil separation test was finally selected because of its simplicity and reproducibility. This test (3), now designated "Cratered Pail Bleeding Test," makes use of screen-supported craters formed by inserting three right circular wire screen cones into the grease, then removing the grease from the cones. The cones are 3 in. in diameter and 4 in. deep and are fabricated from 8-mesh, 0.028-in. diameter wire screen. Three craters are used in each 35-lb test pail. Oil separation is measured by calculating

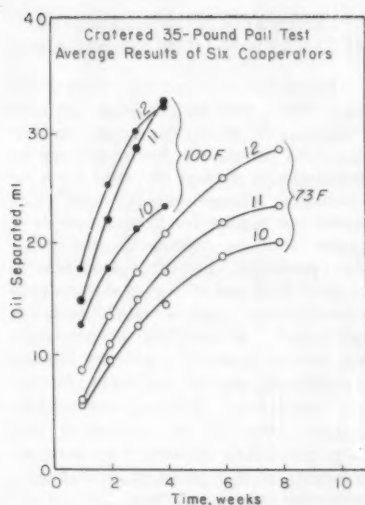


Fig. 7.—Oil separation from test greases.

the average volume of oil in the crater from the surface diameter of the pool of separated oil.

The Cratered Pail Test was evaluated by six cooperators on three greases whose properties are listed in Table VI.

Field experience of the supplier of the three types of greases tested by the committee showed that increasing amounts of oil separated from greases

TG-IV-10, TG-IV-11, and TG-IV-12 in that order. Average results of the Cratered Pail Test rank these greases in the same order, as shown in Tables VII and VIII and Fig. 7. The precision of the Cratered Pail Test results is good. For oil separation times of two weeks at  $73 \pm 4$  F, the per cent standard deviation between the results of different cooperators was 13, 4, and 4, respectively, for the three test greases. Although the above data were obtained on a series

TABLE VI.—PROPERTIES OF TEST GREASES.

Grease.....	TG-IV-10	TG-IV-11	TG-IV-12
Soap type.....	Sodium-calcium	Sodium-calcium	Lithium
Soap, per cent.....	21.7	21.2	17.6
Water, per cent.....	None	None	Trace
ASTM worked penetration <sup>1</sup> .....	260	328	327
ASTM dropping point, ° deg Fahr.....	314	378	370
Oil viscosity, SSU, 100 F.....	166.8	223	66.8
Oil viscosity, SSU, 210 F.....	44.3	47.8	35.3
Oil viscosity index.....	97.8	97.0	50
Oil pour point, deg Fahr.....	-20	-25	Below -55

<sup>1</sup> Method of Test for Dropping Point of Lubricating Grease (D 566-42), 1955 Book of ASTM Standards, Part 5, p. 265.

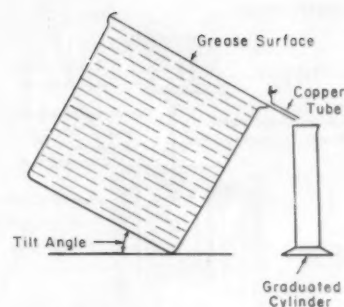


Fig. 8.—Tilted pail bleeding test.

of only three greases, it is believed that the reproducibility of results in these tests justifies the selection of the Cratered Pail Test as a primary storage stability test. Additional tests on a Coordinating Research Council project (report not yet released) confirm this action.

Experience suggests that the reproducibility of data is improved if temperatures do not vary more than  $\pm 2$  F from the values selected and that uniform right circular cones be used.

#### Tilted Pail Tests

In order to reduce the measurement of oil separation tendency to its simplest terms, it was proposed to measure the

TABLE VII.—OIL SEPARATION TESTS IN CRATERED 35-LB PAILS AT ROOM TEMPERATURE. (Milliliters of Oil.)

Time, Weeks	Grease TG-IV-10						Grease TG-IV-11						Grease TG-IV-12					
	1	2	3	4	6	8	1	2	3	4	6	8	1	2	3	4	6	8
California Research Corp. (73 ± 1 F).....	6.7	10.4	14.4	16.0	20.2	20.2	6.9	11.3	16	18.7	25.1	26.9	9.5	13.2	18	20.2	26.9	27.4
General Petroleum Corp. (70 F).....	5.3	8.2	10.8	13.0	18.7	19.7	6.4	10.5	13.4	15.9	20.2	21.7	9.5	13.9	16.4	21.3	25.9	29.2
Union Oil Co. (77 F).....	5	10	12.2	14.2	17.2	20	5	11	15	17.5	20	21.5	7	13	17.5	21	25	29
Average.....	5.7	9.5	12.5	14.4	18.7	20.0	6.1	10.9	14.8	17.4	21.8	23.4	8.7	13.4	17.3	20.8	25.9	28.5
Standard deviation.....	0.9	1.2	1.8	1.5	1.5	0.2	1.0	0.4	1.3	1.4	2.9	3.1	1.4	0.5	0.8	0.6	1.0	1.0
Per cent standard deviation.....	16	13	15	10	8	1	16	4	9	8	13	13	17	4	5	3	4	4

TABLE VIII.—OIL SEPARATION TESTS IN CRATERED 35-LB PAILS AT 100 F. (Milliliters of Oil.)

Time, Weeks.....	Grease TG-IV-10				Grease TG-IV-11				Grease TG-IV-12			
	1	2	3	4	1	2	3	4	1	2	3	4
California Research Corp. old.....	11.5	16.9	19	21.8	15	23.2	29	31.9	15.6	22.4	28	29.6
new.....	13.6	18.7	25.1	25.1	16	23.4	28.8	30.7	13.6	18.7	21.8	20.2
Lefingwell Chemical Co.....	12	15	18	20	16	22	29	37	20	30	38	41
Shell Oil Co. old.....	13	20	24	24	15	19	29	36	(4)	35	39	43
new.....	15	18	22	26	16	28	38	38	23	28	34	34
Tidewater Oil Co.....	11.5	19.2	23.9	27.9	13.4	20.8	25.5	32.6	17.8	24.7	30.6	35.3
Union Oil Co.....	12	16	18	19	13	18	21	24	16	19	21	24
Average.....	12.7	17.7	21.4	23.4	14.9	22.1	28.6	32.9	17.7	25.4	30.3	32.4
Standard deviation.....	1.3	1.8	3.1	3.3	1.3	3.3	5.1	4.3	3.3	6.0	7.2	8.4
Per cent standard deviation.....	10	10	14	14	9	15	18	13	19	24	24	26



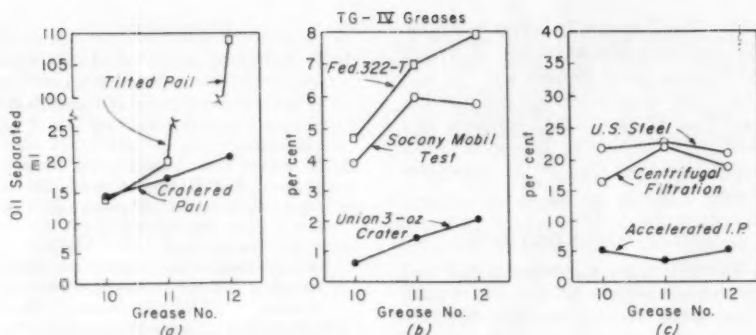


Fig. 9.—Oil separation with various tests.

separation of oil from the smoothed surface of the grease in standard 35-lb pails stored in a tilted position. To facilitate measurement, a draw-off tube was soldered into the apex of the swedge and the separated oil collected in a graduate (see Fig. 8).

As might be expected, the amount of bleeding was found to be roughly proportional to the angle of tilt. For the cooperative phase, an angle of 45 deg was agreed upon. This method showed reasonable reproducibility between laboratories, considering the lack of uniform temperature control, and it evaluated the greases in the same order as in the Cratered Pail Test (see Table IX). In one case, however, the most fluid grease cracked and slumped in the tilted containers.

TABLE IX.—TILTED PAIL TEST RESULTS.

Oil Separation After Four Weeks, Room Temperature, Milliliters of Oil

Grease.....	TG-IV-10	TG-IV-11	TG-IV-12
Laboratory:			
No. 1.....	14	19	118
No. 2.....	5.4	9.6	107
No. 3.....	0.3	20.4	114
No. 4.....	11	16	67
No. 5.....	39	34	138
Average.....	13.9	20	109

#### Accelerated Laboratory Tests

In addition to measuring oil separation in 35-lb pails, each committee member conducted promising accelerated laboratory tests in an effort to develop a small-scale test for determining the storage stability of grease. A brief description of the laboratory tests employed is presented below.

#### Atmospheric Pressure

**Union 3-oz Crater Test (4).**—This test determines the tendency of grease to separate oil during 200 hr storage at 130 F. A sample of grease is placed in a 3-oz tin, 2½ in. in diameter and 1½ in.

\* Method of Test for Apparent Viscosity of Lubricating Greases (D 1092-55), 1955 Book of ASTM Standards, Part 5, p. 566.

high, with a ¼-in. diameter hole in the center of the bottom. A conical crater is formed in the grease, the apex of the cone being the hole in the bottom of the can, and the base being the rim. Extruded oil is caught in a can and weighed.

**Channel Test.**—Grease is stored in a tall form, 1-lb can for extended periods at ambient temperature or 100 F. A cleanly cut channel, 1 in. deep and 0.75 in. wide, is made from side to side in the grease prior to storage. Oil seepage into the channel is measured at monthly intervals.

#### Low Pressure

**Federal Test Method 322-T.**—This method determines the oil separation tendencies of greases when supported on a 100-mesh screen and subjected to constant air pressure of 0.35 psi for 24 hr at 77 ± 2 F.

**Institute of Petroleum Oil Separation Test (5).**—This measures the tendency of grease to separate oil when placed in a metal cylinder having a 60-mesh gauze cone bottom and subjected to a 100-g metal weight for seven days at 25 C (77 F) or 40 C (104 F).

**Accelerated Institute of Petroleum Bleeding Test.**—Same as above, except the grease is subjected to a 185-g metal weight for 24 hr at 125 F (51.5 C).

#### High Pressure

**U. S. Steel Pressure Oil Separation Test (6).**—This procedure measures the tendency of grease to separate oil when placed on a brass screen and subjected to 100 psi nitrogen pressure in an ASTM Apparent Viscosity Cylinder\* for 22 hr at ambient temperatures.

**Centrifugal Filtration Test (3).**—This determines the oil separation which occurs when 1 g of grease is placed in a No. 0000 Coors Büchner funnel and centrifuged for 30 min at approximately 3000 rpm in an International Clinical Centrifuge at ambient temperatures.

**Socony Mobil Pressure Filtration Test (7).**—This determines the amount of oil that separates when lubricating grease is placed in a glass funnel,

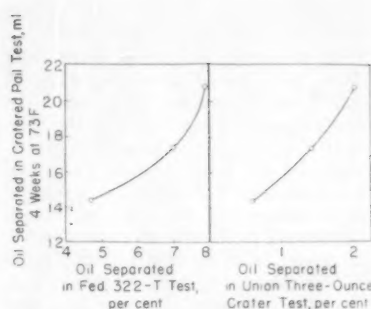


Fig. 10.—Correlation of test results.

equipped with a fritted glass filter plate, and subjected to 7.5 psi air pressure for given periods of time at ambient temperatures.

#### Correlation of Oil Separation Tests

Correlation of the various oil separation tests carried out by Section IV was divided into two parts, the correlation of primary 35-lb pail storage tests with field experience and correlation of accelerated laboratory tests with the 35-lb pail tests.

#### Correlation of Pail Storage Tests with Field Experience

The object of these tests was to select a 35-lb pail test which correlated with service experience. For this purpose, three sample test greases were supplied by one of the committee members whose experience with these products indicated that they had a definite order of oil separation. These three greases were tested at approximately 77 F in the Cratered Pail and Tilted Pail tests, the results being shown in Fig. 9 (a). The order of increased bleeding, as reported by the committee member supplier, was greases TG-IV-10, TG-IV-11, and TG-IV-12.

As can be seen from Fig. 9 (a), the oil separated in both tests in the order expected, although in the Tilted Pail Test grease TG-IV-12 showed oil separation far in excess of that expected from the experience of the supplier. The results of the Cratered Pail Test, however, were about as anticipated. The large oil separation of grease TG-IV-12 in the Tilted Pail Test was probably due to cracking and flow of the rather soft grease. Since the object of these tests was to select a primary storage test which reproduced field experience, the Cratered Pail Test was selected as the primary storage test.

#### Correlation of Accelerated Laboratory Tests with 35-lb Pail Tests

Several accelerated laboratory tests were investigated for their usefulness in estimating oil separation tendencies of the three types of greases used for the

committee tests. The results of these tests are shown in Figs. 9 (b) and 9 (c). Only two tests, the Union 3-oz Crater Test and the Federal Specification 322-T Oil Separation Test, rated the three types of greases used by the committee in the same order as field experience and the 35-lb Cratered Pail Test. Correlation of the laboratory results of these greases with those obtained in the Cratered Pail Test is given in Fig. 10. It should be noted that the only satisfactory laboratory tests are those which used low pressures (1 psi or less) and temperatures fairly close to room temperature.

In some of the pressure tests, packing of the screen or filter paper may have taken place. In tests which rapidly accelerate oil separation (large percentage of oil separates), results can be quite different from those obtained where small percentages of oil are separated during the test period.

### CONCLUSIONS

1. Compressive and tensile forces are important factors in oil separation from greases.

2. In accelerated laboratory tests, pressure and temperature should not deviate markedly from those encountered in bulk storage. Pressure should not exceed 1 psi, and the temperature should not be above 130 F.

3. The Cratered Pail Test is reliable for evaluating the tendency of greases to separate oil in storage, and

4. The 3-oz Crater Test and the Federal Test Method 322-T correlate well with the Cratered Pail Test on the three types of greases used in the committee tests.

### RECOMMENDATIONS

1. Determine the repeatability and the reproducibility of the 3-oz Crater Test and the Federal Test Method 322-T on a variety of greases covering a wide range of consistencies, and

2. Develop an improved laboratory test which requires less time than the 3-oz Crater Test and a smaller sample than the Federal Test Method.

#### Acknowledgments:

Grateful acknowledgment is made to former section members who gave generously of their time and who added much to the progress of the work. These are Earl Amott, Union Oil Co.; D. E. Bowers, General Petroleum Corp.; C. E. Emmons, The Texas Company; R. C. Jones, Shell Oil Co.; L. W. McLennan, Union Oil Co.; and L. Mittelman, Tidewater Oil Co.

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## Polyethylene Environmental Stress Cracking by the Proposed Bent Strip Test Method

By K. A. KAUFMANN<sup>1</sup>

*Test is useful for product comparison and inspection but lacks interlaboratory reproducibility.*

**S**INCE November, 1954, the bent strip method of test for environmental stress cracking of ethylene plastics has been under study by Subcommittee XV on Thermoplastic Materials, of ASTM Committee D-20 on Plastics. The Task Group responsible for studying this method under that subcommittee has

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not yet been able to control all the variables of the method to secure adequate between-laboratory reproducibility of results, although within-laboratory accuracy is fair. A previous reporting (1)<sup>2</sup> indicated the status of this work as of the fall of 1956.

The Task Group's original assignment

<sup>2</sup> The boldface numbers in parentheses refer to the list of references appended to this paper.

<sup>3</sup> Tentative Specification for Polyethylene Molding and Extrusion Materials (D 1248-52 T), 1955 Book of ASTM Standards, Part 6, p. 109. (Note: Specification D 1248 was revised in 1958 and will appear in the 1958 Book of ASTM Standards, Part 9.)

was to develop this proposed method of test for use as an identification of ethylene polymers for incorporation in the ASTM Specification for Polyethylene Molding and Extrusion Materials (D 1248-52 T).<sup>3</sup> In November, 1957, the Task Group summed up the results of its work as follows:

"The Task Group does not yet have an environmental stress cracking test procedure capable of giving precise, universally reproducible  $F_{50}$  values [time at which half the specimens have failed]. Major causes of this appear to be the problems of reproducibly preparing test sheets, standardizing the geometry of the test

specimen under stress, and accounting for creep relaxation of the specimen. The bent strip procedure, as published for information in the February, 1957 Compilation of ASTM Standards on Plastics, may be useful for routine inspection and acceptance tests. The Task Group recommends that only this form of the procedure be considered for inclusion in the pending revision of the polyethylene materials specification D 1248 and that consideration of  $F_{50}$  times be deferred until a satisfactory solution to the problem is found.<sup>1</sup>

The proposed method is not presently included in Tentative Specification D 1248, but is published in proposed form only, having no official status. A somewhat improved version is being published in the 1958 Compilation of ASTM Standards on Plastics. This revision still lacks interlaboratory reproducibility and is recommended only for routine inspection and acceptance testing.

By "routine inspection and acceptance testing" is meant that individual laboratories must establish the level of acceptability of materials solely on the basis of test results within their own laboratory with full realization that other laboratories may not obtain similar results with the same lot of material.

#### History of Test Work

The first two round robin test programs have been adequately summarized in previous publications (1). Both used essentially the method described. The data obtained from the first two programs are not shown here, but excerpts were published in the previous articles (1).<sup>2</sup> It was seen that a very definite lack of reproducibility between laboratories existed; but that, generally duplicate runs within a given laboratory gave fair reproducibility. All materials tested were normal den-

sity polyethylenes with the exception of sample R-1 which was a higher density (0.96) polymer. Present ASTM density classifications are 0.910 to 0.925 (type I), 0.926 to 0.940 (type II) and 0.941 to 0.965 (type III).

This proposed method of test was originally developed by the Bell Telephone Laboratories in Murray Hill, N. J., (2,3) and that procedure was utilized in the first round robin test. The only basic difference between that and the previously referenced ASTM published procedure was the use of a test tube alone to hold the specimens instead of the channel jig now specified. The second round robin test utilized these channel specimen holders and investigated the effect of dilute reagent, specimen preparation variables, and bending jigs. It was concluded that dilute reagent was not useful for classifying the resins studied under the specific test conditions used. Specimens prepared at one location gave better among-laboratory agreement than those prepared at the testing site, and it was thus concluded that specimen preparation was one of the important uncontrolled variables. From results of two laboratories testing both with and without mechanical bending jigs to control this variable, it was concluded that this too was an important variable.

In an attempt to determine the effect of specimen molding conditions upon tests results, a study of the conditions used by each participating laboratory to produce the specimens for the second round robin was made. No correlation was found.

#### Third Round Robin

The basic procedure for the proposed method was first revised on August 9, 1956. This revision allowed closer control of the molding cycle, specified a window or chase mold, flash type, with thin plates instead of the standard ASTM compression mold, and ruled out the use of chemical release agents during molding (because many of these are

effective stress crack agents). These changes were based on evidence of their need. This is the procedure being published in the 1958 Compilation of ASTM Standards on Plastics.

The August 9, 1956, procedure was called procedure A for the third round robin and the following variations from it were also studied:

*Procedure B.*—Fast cooling during sheet molding (2 min instead of 8).

*Procedure C.*—Use of 0.075-in. thick specimens (instead of 0.125 in.).

*Procedure D.*—Use of 20 per cent reagent solution by weight in water instead of concentrated; and test at 23 C instead of 50 C.

*Procedure E.*—Use of the Bell Telephone Laboratories' bending and transfer jigs.<sup>4</sup>

The three materials used were selected from among those used in the previous programs. A sample of the data is shown in Table I.<sup>5</sup> It was concluded that agreement between laboratories was still less than satisfactory in all procedures, although each variation on the basic procedure seemed to improve the situation. These improvements, however, did not always rank the resins in the same order of acceptability. Procedures C and D appeared best of the variations tried. Extra work done by some laboratories indicated some advisability of combining some of the variations into a single test and a strong necessity for use of higher annealing temperatures with increasing specimen density.

#### Fourth Round Robin

Accordingly, it was decided that a fourth round robin should be conducted using a method based upon procedure A from the third round robin as modified by procedures C, D, and E (0.075 in. specimens, 20 per cent reagent and Bell Telephone Laboratories' bending and transfer jigs). Although the use of these jigs indicated no greater improvement than the fast cooling procedure B, they were preferred because of the mechanical ease of manipulation they provided to the test and because the fast

TABLE I.—THIRD ROUND ROBIN ON STRESS CRACKING OF POLYETHYLENE.  
Hours for 50 per cent failure, Sample K-3, Procedures A-E, 12 participating laboratories.

Procedure . . . .	A			B			C			D			E		
Run . . . . .	1	2	Average	1	2	Average	1	2	Average	1	2	Average	1	2	Average
	13	10	11	2.2	5.3	3.8	>216	>216	>216	96	43	69.5	102	144	123
	14.0	12.0	13.0	144	156	150	>312	>312	>312	210	200	205	11	73	42
							>336	>336	>336				>336	>336	>336
							>336	>336	>336				81	96	88.5
	23	165	20	190	98	144	>360	>360	>360	122	110	116	>336	>336	>336
	110	>336	57.5							72	100	86	34	34	34
	35	80								>336	>336	>336	>336	>336	>336
	>336	>336	>336	145	175	160							>336	>336	>336
	110	91	100	25	40	32.5							>336	>336	>336
	25	75	50			5.0									
			9.75												3.5



cooling (2 min) might not be universally attainable because of equipment limitations. In addition, a technique for annealing higher density resins at higher temperatures was adopted as follows:

Type	Density Range, $d^{25}$	Temperature, deg Cent
I.....	0.910 to 0.925	100 $\pm$ 1.0
II.....	0.926 to 0.940	110 $\pm$ 1.0
III.....	0.941 to 0.965	120 $\pm$ 1.0

In this fourth round robin, two of the lower density resins and the one higher density material from the previous round robins were used plus a medium density (0.935) resin made by the high-pressure process. Three methods of higher temperature annealing were employed: air oven annealing followed by quick cooling at room temperature, as the basic procedure; air oven annealing followed by slow cooling in the oven to room temperature; and, autoclave annealing followed by quick cooling at room temperature. Annealing time was 1 hr in all cases.

A sample of the resulting data is shown in Table II and it is readily apparent that interlaboratory agreement was not attained.<sup>5</sup> At this point, the Task Group decided not to conduct further cooperative programs until more definite ideas could be developed to improve the situation. In addition to work being done on the bent strip test by a number of laboratories individually,

<sup>5</sup> Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618-54), 1955 Book of ASTM Standards, Part 6, p. 447; Tentative Revision of Methods D 618, 1956 Supplement to Book of ASTM Standards, Part 6, p. 353.

a few are investigating other means of measuring this property.

#### Industry Status

The proposed method is being widely used in industry as a "go-no-go" gage for product comparisons and inspection acceptance practices. Specifically, its use in this area has been to allow not more than 20 per cent failure of specimens in 48 hr for type I polyethylene materials. Such a criterion has been used by the Bell Telephone Laboratories for about 10 years in their specifications for type I polyethylenes of low melt index ( $<0.5$  g per 10 min) for cable sheathing. Essentially the method used is the procedure of 1956 plus the Bell Telephone Laboratories bending and transfer jigs as shown in the 1958 Compilation of ASTM Standards on Plastics.

In addition, those laboratories concerned with medium- and high-density polyethylenes generally anneal specimens for 1 hr at the temperatures discussed above in air ovens or autoclaves in place of boiling water temperatures. They are then equilibrated at  $23 \pm 1$  C (ASTM Methods D 618-54)<sup>6</sup> and tested within 24 hr.

#### Acknowledgment:

The contributions of the following members of the Task Group in performing the extensive work in all the round robins, in the interpretation of the data, and in guidance of the program, represent a monumental effort without which this test would be an unknown entity. The plastics industry should be grateful to this group for their efforts as is ASTM Committee D-20 on Plastics.

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TABLE II.—FOURTH ROUND ROBIN ON STRESS CRACKING OF POLYETHYLENE.  
Hours for 50 per cent failure, 6 participating laboratories.

Sample.....	A-3			K-3			R-1			X-1		
	1	2	Average	1	2	Average	1	2	Average	1	2	Average
Run. ....												
	3.0	2.8	2.9	9.4	6.9	8.2	165	160	162.5	3.8	4.0	3.9
	220	220	220	336	336	336	290	300	295	310	321	216
	3.0	2.7	2.9	30	29	29.5		See Note 1		0	0	0
	200 <sup>a</sup>	$>336^a$		$>336^a$	$>336^a$	$>336^a$	$>336^a$	$>336^a$	$>336^a$	$>336^a$	$>336^a$	$>336^a$
	25	25	25	47	51	49	178	190	184	0.85	0.85	0.85
	21.5	27	24	31	30	30.5	200	200	200	24	30.5	37

<sup>a</sup> Did not use bending jig.

NOTE 1.—Cold drawing occurred on bending, normal failures did not occur, failures at outer edge: after 170 hr

Standard Procedure		Slow Cooling	
1	2	1	2
60 per cent	10 per cent	100 per cent	20 per cent

# Thermal Stability of Glasses in ASTM Thermometers

By ERNEST L. RUH and GEORGE E. CONKLIN

Measurement of bulb and stem changes in thermometers from 14 manufacturers reveals variation in their stability at high temperatures. Comparative data on special thermometers indicate possibilities for more stable instruments. Shortcomings are noted in the ASTM Permanency of Range Test and in ASTM limitations for high-temperature use.

**L**IQUID-IN-GLASS thermometers comprise a relatively inexpensive series of instruments for use in laboratories dealing with the examination of petroleum and other products where ready and convenient means for temperature measurement is important. Such instruments have found adaptation in a variety of types having temperature ranges within the approximate limits of -100 and +1000 F. The boundaries of this interval are determined largely at the lower limit by the availability of suitable liquids and at the upper limit by the softening characteristics of the glasses used. The manufacturing requirements for most of these instruments are included in the Standard Specifications for ASTM Thermometers.<sup>1</sup> Such thermometers do not exceed an upper range limit of 760 F, and the glasses to be used are defined as follows:

"The stem shall be made of suitable thermometer tubing. . . ."

"The bulb shall be made of Corning normal, Kimble R 6, or equally suitable thermometric glass."

These general definitions would be wholly inadequate to insure users that the glasses are "suitable" except for a performance test that is required for most, but not all, ASTM thermometers scaled to 580 F or higher. The performance test is known as the Test for Permanency of Range and was first proposed by ASTM in 1921.<sup>2</sup> "The purpose of the provision is to furnish a means for determining that the ther-

monometer has been adequately annealed so that subsequent changes in bulb volume will not be excessive."<sup>3</sup>

The general requirements for ASTM thermometers<sup>1</sup> also include the note "Thermometers made with these bulb glasses (defined above) should not be subjected to temperatures above 760 F or be continuously exposed to temperatures above 700 F." It is common knowledge that under the pressure of circumstances this warning has often been disregarded.

The intent of the investigation reported in this paper was twofold. First it was desired to throw some light on the degree of attention given to proper annealing in present-day manufacturing techniques. Second, the magnitude and nature of the errors resulting from the use (or misuse) of improperly annealed thermometers was considered to be of interest. Other conclusions drawn from the data obtained are believed to be of value.

## Discussion

Based on the expansion characteristics of mercury, a Fahrenheit mercury-in-glass thermometer has a bulb volume equivalent to 11,000 times the

volume of one degree of stem length. The volume of the capillary in a thermometer being relatively small, the changes due to exposure to high temperatures can be expected to produce proportionately small scale errors compared to those arising from changes in the bulb. The ASTM Test for Permanency of Range (Bulb Stability) would be limited to bulb change measurements if immersion of thermometers in the bath could be confined to the bulbs. In this investigation the bulbs and a portion of the stems were exposed. Consequently, data are presented to differentiate between the changes.

When thermometers are heated to high temperatures and then cooled, both temporary and permanent changes may take place.<sup>4,5</sup> The former will be manifested as relatively small ice-point depressions which should almost entirely disappear in time. The latter or permanent changes can be large if proper annealing has not been employed. In such cases, the strains present in the glasses are relieved to some extent at the high temperature. The glasses contract and on cooling permanent elevation of ice point and all other readings along the scale will result. Upon successive reheating and cooling, these changes will continue indefinitely.

The changes discussed above are common to all thermometers, but normally would not exceed 0.2 F for

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<sup>1</sup> Specifications for ASTM Thermometers (E 1-58), 1958 Book of ASTM Standards, Parts 4, 7, and 8.

<sup>2</sup> Report of Subcommittee VIII on Distillation of Committee D-2 on Petroleum Products and Lubricants, *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 367 (1921).

<sup>3</sup> E. F. Mueller and R. M. Wilhelm, "Methods of Testing Thermometers," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 500 (1938).

<sup>4</sup> H. C. Dickinson, "Heat Treatment of High-Temperature Mercurial Thermometers," *NBS Scientific Paper 32*, Nat. Bureau Standards, April, 1906.

<sup>5</sup> Testing of Thermometers, *NBS Bulletin No. 8*, Nat. Bureau Standards, 2nd Edition (1911).



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thermometers sealed or used below 300 F.<sup>6</sup> For thermometers employed at higher temperatures, such changes may amount to many degrees.

### Outline of Investigation

A type of thermometer was chosen for this investigation that should reflect maximum changes encountered with ASTM thermometers. Special thermometers of this type were included also that were expected to represent the ultimate or most stable instruments that could be fabricated.

ASTM High-Distillation Thermometers, ASTM No. 8 F, range 30 to 760 F were purchased from sources representing 14 manufacturers located in the northeastern section of the United States. Eleven of these makers used Corning normal and three used Kimble R6 glass for the bulbs. All used Corning lead tubing for the stems. In addition, three special thermometers were fabricated specifically for this investigation. These are described in Table I.

TABLE I.—EXPERIMENTAL THERMOMETERS.

Special Experimental Thermometers, Designation	Description
O.....	Made to ASTM No. 8 F requirements with Corning lead stem and Corning normal bulb glasses, but with bulbs compensated for anticipated change by placing a calculated amount of glass inside each bulb. <sup>a</sup>
P.....	Made to ASTM No. 8 F requirements, but with stems and bulbs fabricated from borosilicate glass.
Q.....	Made to ASTM No. 8 F requirements, but with stems and bulbs fabricated from Corning 172 glass.

<sup>a</sup> See Patent No. 2,578,563 granted Dec. 11, 1951, to L. C. Liberatori and assigned to the Taylor Instrument Companies.

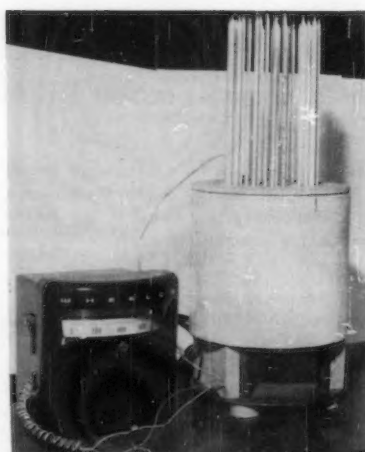


Fig. 1.—Permanency of range (bulb stability) bath—aluminum block type.

A total of 68 thermometers were used, four from each of the 14 manufacturers A to N and four each of the three special thermometers O, P, and Q. Accurate readings at 32.0 and 210.0 F were first observed and recorded for each individual thermometer. The entire lot

was then divided into two equal groups. One group was subjected to successive periods of exposure at 700 F and the other at 760 F.

The group of thermometers subjected to 700 F were exposed for the periods indicated in Table II for a total of 600 hr. The group subjected to 760 F were exposed for the periods indicated in Table III for a total of 350 hr. In all cases exposure to the elevated temperatures was made in the aluminum block (insulated) bath shown in Fig. 1, which conforms to the one illustrated in Fig. 2 of ASTM Method E 77.<sup>7</sup> Immersion for both groups approximated the 150 F graduation lines. Temperatures were controlled within  $\pm 2.0$  F in all instances. Except where otherwise stated, ice-point readings were made 30 to 60 min after removal of the thermometers from the bath.

<sup>6</sup> Johanna Busse, "Liquid in Glass Thermometers," Temperature—Its Measurement and Control in Science and Industry, Reinhold Publishing Corp., New York, N. Y., p. 231 (1941).

<sup>7</sup> Method of Testing and Standardization of Etched-Stem Liquid-in-Glass Thermometers (E 77-58), 1958 Book of ASTM Standards, Part 7.

TABLE II.—BULB STABILITY ASTM THERMOMETER No. 8 F AT 700 F.

Thermometers		Ice-Point Change at the End of Various Hours Exposure, deg Fahr										
		24	48	72	96	168	240	312	384	456	528	600
Manufacturer	A.....	1.8	3.6	4.4	6.0	8.0	10.0	11.7	12.3	13.8	14.3	16.0
	B.....	0.0	1.2	2.0	2.8	4.3	6.2	7.3	8.6	9.1	10.0	11.2
	C.....	3.1	4.7	4.8	5.3	6.2	7.7	8.6	9.1	9.7	9.9	11.3
	D.....	2.1	3.2	3.9	4.1	5.3	5.9	6.2	6.9	7.3	7.6	8.1
	E.....	2.1	2.4	3.2	3.6	4.5	5.2	6.6	6.8	6.9	7.7	7.9
	F.....	1.2	1.8	2.2	2.7	3.0	4.0	4.5	4.6	4.7	4.9	5.9
	G.....	1.8	2.1	2.9	3.1	3.5	4.0	4.1	4.5	4.7	5.0	5.8
	H.....	0.1	0.7	1.7	1.8	2.0	2.2	3.1	3.1	3.3	3.7	5.0
	I.....	0.5	1.5	1.7	1.8	2.5	2.5	3.5	3.6	3.6	3.6	3.8
	J.....	-0.1	0.1	0.5	0.9	1.5	2.0	2.0	2.5	3.0	3.3	3.8
	K.....	-0.3	-0.2	0.5	1.0	1.6	1.4	1.4	1.6	1.6	1.8	2.6
	L.....	0.1	0.2	0.6	0.9	0.8	1.7	1.8	1.8	1.9	1.9	2.6
	M.....	0.1	0.1	0.6	0.8	1.2	1.5	1.5	1.7	1.9	1.9	2.1
	N.....	0.0	0.0	0.0	0.0	0.0	0.1	0.1	0.2	0.2	0.2	0.3
	Special	O.....	0.1	0.1	0.0	0.0	-0.1	0.0	-0.1	-0.1	-0.1	-0.1
Thermometers	P.....	0.0	0.0	0.0	0.1	0.0	0.1	0.0	0.1	0.1	0.1	0.0
	Q.....	0.0	-0.1	-0.1	-0.1	-0.2	-0.1	-0.2	-0.2	-0.2	-0.2	-0.1

NOTE.—All values represent average for two thermometers.

TABLE III.—BULB STABILITY ASTM THERMOMETER No. 8 F AT 760 F.

Thermometers		Ice-Point Change at End of Various Hours Exposure, deg Fahr																			
		0.5	1.0	1.5	2.0	3	6	9	12	15	18	21	24	27	30	50	75	100	200	350	
Manufacturer	A.....	-2.0	-1.8	-1.0	-0.3	0.2	0.8	1.8	3.0	4.4	5.5	6.5	7.2	8.1	8.9	12.4	14.8	16.4	20.9	23.8	
	B.....	-2.3	-2.2	-2.2	-1.8	-1.8	-1.6	-1.3	-0.2	0.6	1.1	2.2	2.6	3.1	4.0	6.7	9.3	10.0	15.1	18.8	
	C.....	2.0	2.0	2.0	2.4	3.6	4.0	4.0	4.2	4.6	5.8	5.8	5.8	6.2	7.0	8.4	10.2	12.0	15.0	17.8	
	D.....	0.0	0.1	0.2	1.0	1.0	1.8	2.1	2.6	3.5	3.8	3.9	4.2	4.3	4.4	5.9	7.3	7.9	10.2	11.9	
	E.....	-0.2	-0.2	-0.2	1.0	1.5	2.0	2.0	2.5	3.0	3.0	3.0	3.1	3.5	4.0	4.7	5.5	6.1	8.5	11.3	
	F.....	-1.3	-1.3	-1.1	-1.0	-0.2	0.4	0.5	0.7	0.8	1.4	2.2	2.3	2.6	3.0	4.2	5.8	6.5	10.0	12.2	
	G.....	-0.2	0.0	0.2	0.5	1.2	1.4	1.6	1.8	2.2	2.4	2.5	2.8	3.0	3.2	4.2	5.0	5.3	7.0	7.6	
	H.....	-0.1	-0.1	0.0	-0.1	0.0	0.0	0.0	0.0	0.1	0.4	1.0	1.3	1.8	2.0	2.1	3.5	4.2	6.5	7.9	
	I.....	-0.5	-0.4	-0.3	-0.4	-0.3	-0.2	-0.2	-0.2	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	0.5	0.9	1.8	2.8	3.8	
	J.....	-0.3	-0.3	-0.4	-0.4	-0.4	-0.3	-0.3	-0.3	-0.3	-0.2	0.0	0.4	0.4	0.9	1.9	2.6	3.2	5.5	7.3	
	K.....	-0.9	-0.9	-0.9	-0.9	-0.9	-0.9	-0.9	-0.9	-0.9	-0.7	-0.6	-0.3	-0.1	0.1	0.1	1.1	1.2	2.3	3.1	3.4
	L.....	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.3	0.4	0.6	0.6	1.0	1.0	1.7	2.0	2.1	2.2	2.4	
	M.....	-0.3	-0.1	-0.1	-0.1	0.0	0.1	-0.1	-0.1	-0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.3	0.4	0.4	1.4	2.0
	N.....	-1.2	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-1.1	-0.6	-0.5	-0.5	0.2	0.4
	Special Thermometers	O.....	-0.2	-0.2	-0.2	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1	-0.3	-0.3	-0.3	-0.4	-1.6
P.....		0.0	0.1	-0.1	-0.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.4	0.6	0.6	
Q.....		0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	-0.1	-0.1	

<sup>a</sup> Values for C represent worse of two thermometers. All other values represent average for two.



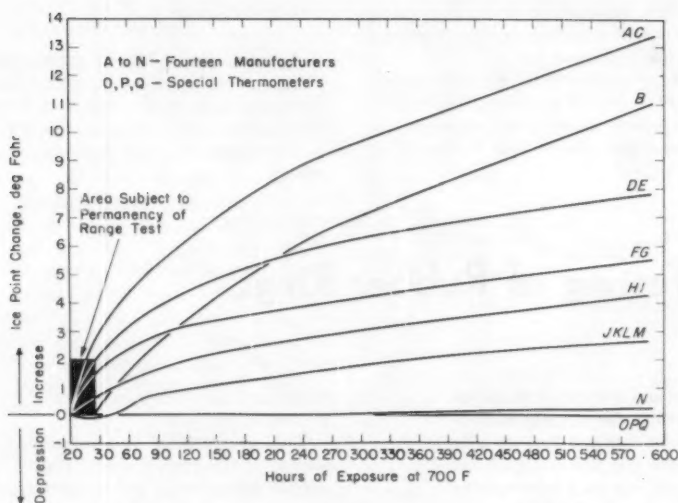


Fig. 2.—Bulb stability—ASTM thermometer No. 8 F at 700 F.

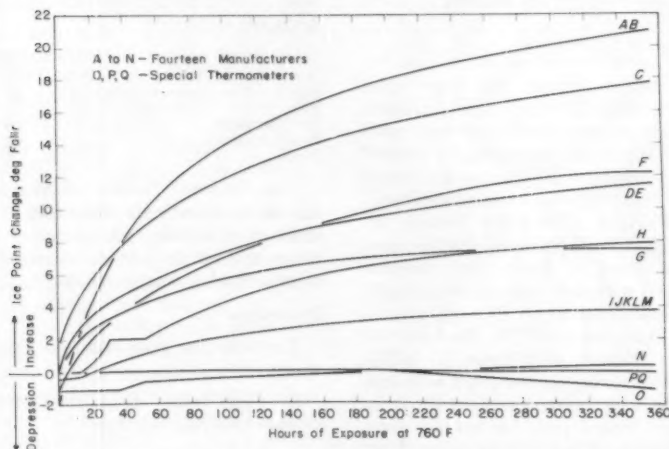


Fig. 3.—Bulb stability—ASTM thermometer No. 8 F at 760 F.

### Observations

Figures 2 and 3 present graphically the ice-point changes observed after exposure. Changes of similar type and magnitude were averaged and indicated by a single curve where appropriate. In general, increases in ice point were uniform and in direct proportion to temperature and length of exposure. No trend was observed to indicate a superiority for either Corning normal or Kimble R6 glass for use in bulbs.

In Fig. 2 the rectangle comprising the area subject to Permanency of Range Test discloses that thermometers representing manufacturers A, C, D, and E would either fail or be questionable. Curve B represents the unreliable situation where an apparently stable thermometer would fail rapidly after passing the test with no change. The curve for J, K, L, and M represents thermometers reasonably suitable

for the short periods of exposure to high temperatures experienced in ASTM tests. Curve N reveals that a thermometer of exceptional stability is possible. However, even here a continuous bulb change with time is demonstrated which confirms the caution that such thermometers should never be used continuously at temperatures above 700 F. A lower limit than 700 F probably should apply.

Figure 3 shows ice-point depression to be greatly influenced by the higher temperature; however, the irregularity of occurrence and magnitude conforms with the experience of other investigators.<sup>3</sup> Curve C represents one thermometer which demonstrated exceptional bulb instability during a short exposure at 760 F. Any ice-point depression was overshadowed by bulb shrinkage on this initial exposure to high temperature.

The thermometers represented by curves A and B displayed considerable ice-point depression, but demonstrated even greater bulb instability than curve C for a long period of exposure.

Table IV has been included to indicate the presence or absence of volume changes in the stem capillaries. For most of the thermometers, the values (0.5 or lower) are considered insignificant. For the remainder, stem changes equivalent to approximately 1 F are indicated.

The superior nature of thermometers made from Corning 172 glass is indicated in curves Q. Borosilicate, curve P, is only slightly inferior at 760 F. Thermometers made with compensated bulbs showed no bulb change for 500 hr at 700 F and 200 hr at 760 F. This good performance suggests another possible means for making stable high-temperature thermometers.

TABLE IV.—DATA REVEALING PRESENCE OR ABSENCE OF STEM CHANGES AFTER EXPOSURE TO 760 F FOR 350 HR—IMMERSION TO 150 F GRADUATION LINE ASTM THERMOMETER No. 8 F.

Thermometers	Ice-Point Change	210 F Change	Stem Change, Ice Point Minus 210 F Change
Manufacturer A...	25.9	25.4	0.5
B...	20.7	22.2	-1.5
C...	16.6	15.9	0.7
D...	13.3	12.9	0.4
E...	10.8	11.2	-0.4
F...	13.9	13.6	0.3
G...	8.9	8.9	0.0
H...	9.5	9.5	0.0
I...	5.2	5.0	0.2
J...	10.1	9.0	1.1
K...	5.1	5.1	0.0
L...	3.6	2.3	1.3
M...	2.1	2.6	-0.5
N...	1.2	1.9	-0.7
Special O...	-1.2	-1.1	-0.1
Thermometers P...	1.7	1.9	-0.2
Q...	0.0	0.2	-0.2

NOTE.—All values represent average for two thermometers.

### Summary

The variation in stability of 14 makes of high-distillation thermometers tested at 700 and 760 F was demonstrated by ice-point increases ranging from less than 2 to more than 20 F. The necessity for better annealing practice on the part of some manufacturers is evident.

Using proper annealing practice a thermometer stable for the short term exposures at high temperatures required for ASTM tests can be produced from glasses normally employed.

Abnormal bulb changes observed in this investigation could not be attributed to the use of either Corning normal or Kimble R6 glass, both of which were represented.

Thermometers with compensated bulbs or from Corning 172 or borosilicate glasses might be applied for long exposures at high temperatures where unusual bulb and stem stability is necessary.

The ASTM Permanency of Range Test (Bulb Stability) may yield results

that are misleading or may approve thermometers which it has rendered unfit for continued use.

The precaution in the general requirements for ASTM thermometers<sup>1</sup> advising against continuous exposure to temperatures above 700 F should be modified.

#### Acknowledgment:

Appreciation is hereby expressed for the excellent work and cooperation of those members of the Standard Inspection Laboratory staff who have assisted in the accumulation of data used in this report.

## Tension Testing of Rubber Rings

By F. L. ROTH and R. D. STIEHLER

The effect of type of roller grip on the stress and elongation at failure of rubber bands and rings was investigated at the request of Subcommittee X, of ASTM Committee D-11 on Rubber and Rubber-Like Materials. It was found that the type of roller grip had no significant influence on these properties when castor oil was used between the specimen and roller. Low values may be obtained if a lubricant is not used between the specimen and the roller. It is recommended that lubrication be required in standard methods of test for ring specimens.

IN THE DEVELOPMENT of a method for tension testing of rubber O-rings, Subcommittee X of ASTM Committee D-11 on Rubber and Rubber-Like Materials found that the description of the grips varied in the several methods for tension testing of rubber rings. For example, ASTM Method D 412 - 51 T<sup>1</sup> states: "For testing ring specimens the grip shall consist of two rollers having a minimum diameter of  $\frac{3}{8}$  in. and a maximum length of  $\frac{1}{2}$  in. mounted on ball bearings. The action of the rollers shall be such that as tension is applied they will rotate freely so as to equalize the stress around the periphery of the specimen." Similarly, Federal Standard No. 601 states: "For a ring specimen, each grip consists of two rollers, each with a minimum diameter of  $\frac{1}{4}$  in. and a maximum length of  $\frac{1}{2}$  in. mounted on ball bearings. The action of these rollers is such that as tension is applied they rotate freely in opposite directions, thus tending to equalize the stress around the periphery of the specimen." Both methods require lubrication of the portion of the ring which passes over the pulleys; ASTM Method D 412 specifically requires castor oil. On the other hand Recommendation R 37 of the International Organization for Standardiza-

tion states: "Rings are fitted over pulleys, either 25 mm or 1 in. diameter, both free to rotate and one at least, preferably the lower, automatically rotated by the machine to equalize the strain in the ring while it is being stretched." MIL-P-5315A (1951) for O-rings requires that: "all pull tests shall be conducted using a Scott test machine equipped with a driven (rotating) Scott ring test attachment or equivalent." The Tentative ASTM Method for Tension Testing of O-Rings (D 1414)<sup>2</sup> also utilizes single-roller grips with one power-rotated and describes them as follows: "Spool jaws for testing rings shall consist of ball-bearing rollers  $\frac{1}{2}$  in. in diameter each, and capable of being brought within 0.7 in. center-to-center distance at closest approach. The roller mounted on the power-driven jaw shall be arranged to rotate one full

revolution for each 6 in. of travel of the power-driven jaw."

This situation led to a comprehensive survey of the grips employed in various rubber laboratories and their method of operation. Among 240 laboratories that responded to a questionnaire, 45 make tests on ring specimens. The following grips are employed:

GRIPS	LABORATORIES
Single roller, power-rotated . . . . .	22
Single roller, free to rotate . . . . .	12
Double roller . . . . .	4
Half cylinder . . . . .	3
Hook . . . . .	4

The National Bureau of Standards agreed to investigate the effect of the single and double roller grips on the stress and elongation at failure of O-rings and other ring specimens.

#### Procedure

The following grips were used in the study:

1. Single-roller,  $\frac{1}{2}$  in. in diameter, mounted on ball bearings,
  - (a) Lower roller rotated once per 6 in. travel, and
  - (b) Neither roller power-driven.
2. Double-roller,  $\frac{5}{16}$  in. in diameter, mounted on ball bearings.



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<sup>1</sup> Tentative Method of Tension Testing of Vulcanized Rubber (D 412 - 51 T), 1955 Book of ASTM Standards, Part 6, p. 1129.

<sup>2</sup> Tentative Method for Tension Testing of Rubber O-Rings (D 1414 - 56 T), 1957 Supplement to Book of ASTM Standards, Part 6, p. 229.

3. Double-roller,  $\frac{1}{2}$  in. in diameter, mounted on ball bearings.

An Instron tester with a crosshead speed of 20 in. per min was used in these tests. The stress and elongation at failure were determined from the record on the strip chart, the inside diameter of the ring, and its cross-sectional area. The cross-sectional area was determined from the weight, specific gravity, and inside diameter of the specimen.

Lubrication between rubber and roller was not used in most instances. When significant differences in the values for stress and elongation at failure were obtained with different grips, additional tests were made using castor oil as lubricant.

The ring specimens employed are described in Table I.

### Test Results

Tables II and III summarize the results obtained for the rubber bands and O-rings. The values for stress and elongation at failure represent the mean for 30 specimens of rubber bands and 15 specimens of O-rings. The columns headed "Limits" give the 95 per cent confidence limits for the mean; that is, the mean  $\pm$  the limit value represents the range within which the true mean is likely to be.

It is seen that only in the case of O-rings made from chloroprene rubber (CR) compound does the grip have a significant effect on the values for stress and elongation at failure. In this case, the single roller grips when not rotated give low values and the breaks occur near one of the rollers. However, these low values are not obtained when castor oil is used as a lubricant between specimen and roller.

Table IV gives the results obtained with rings cut from standard sheets prepared in accordance with ASTM Methods D 15 - 57 T,<sup>2</sup> formula 1A for natural rubber compounds, and cured 40 min at 140 C. Each value is the mean for 16 specimens. If no lubricant is used, low values are obtained with all grips, the greatest effect being obtained with single rollers that are not rotated and the least with the same rollers when they are rotated. Castor oil lubrication between specimen and roller eliminates any effect of grip within the precision of the experiment and gives significantly higher values for stress and elongation at failure for all grips.

### Conclusions

The type of roller grip used for testing any ring specimen is not important if castor oil or other suitable lubricant is

<sup>2</sup> Tentative Methods of Sample Preparation for Physical Testing of Rubber Products (D 15 - 57 T), 1957 Supplement to Book of ASTM Standards, Part 6, p. 186.

TABLE I.—COMPOSITION AND DIMENSIONS OF RING SPECIMENS.

Specimen	Rubber and Elastomer Designations <sup>a</sup>	Nominal Dimensions	
		Inside Diameter	Cross-Section
Rubber band.....	NR <sup>b</sup>	2½ in.	¼ × ¼ in.
O-rings.....	NR	2 in.	¾ in. diam
O-rings.....	SBR	2 in.	¾ in. diam
O-rings.....	NBR	2 in.	¾ in. diam
O-rings.....	NBR	1½ in.	¾ in. diam
O-rings.....	NBR	1½ in.	¾ in. diam
O-rings.....	CR	2 in.	¾ in. diam
Cut rings.....	NR	36 mm.	2 × 2 mm

<sup>a</sup> ASTM Tentative Recommended Practice for Nomenclature for Synthetic Elastomers and Latexes (D 1418 - 56 T), 1956 Supplement to Book of ASTM Standards, Part 6, p. 201.

<sup>b</sup> Natural rubber.

TABLE II.—TENSILE STRENGTH OF BANDS AND O-RINGS.

Specimen			Tensile Strength, psi					
Type	Rubber and Elastomer Designations	Inside Diameter, in.	Single Roller Grip				Double Roller <sup>a</sup> Grip	
			Rotated		Free		Mean	Limits
			Mean	Limits	Mean	Limits		
Band.....	NR	2½	2090	140	1980	140	2020	140
O-ring.....	NR	2	2000	250	2110	100	1930	310
O-ring.....	SBR	2	1600	110	1560	80	1540	70
O-ring.....	NBR	2	1610	50	1550	40	1550	50
O-ring.....	NBR	1½	1570	40	1580	50	1620	20
O-ring.....	NBR	1½	1740	30	1680	80	1740	40
O-ring.....	CR	2	2550	100	2030	110	2510	120
O-ring <sup>b</sup> .....	CR	2	2520	90	2490	150	2530	150

<sup>a</sup> ½-in. rollers except for 1½ in. O-rings where ¾-in. rollers were used.

<sup>b</sup> Castor oil used as lubricant between specimen and roller.

TABLE III.—ULTIMATE ELONGATION OF BANDS AND O-RINGS.

Specimen			Elongation, per cent					
Type	Rubber and Elastomer Designations	Inside Diameter, in.	Single Roller Grip				Double Roller <sup>a</sup> Grip	
			Rotated		Free		Mean	Limits
			Mean	Limits	Mean	Limits		
Band.....	NR	2½	690	12	680	11	690	13
O-ring.....	NR	2	310	70	320	30	300	80
O-ring.....	SBR	2	170	9	170	7	170	6
O-ring.....	NBR	2	220	7	210	6	210	8
O-ring.....	NBR	1½	250	8	240	12	250	8
O-ring.....	NBR	1½	240	8	230	13	250	11
O-ring.....	CR	2	230	12	190	11	230	11
O-ring <sup>b</sup> .....	CR	2	230	14	230	13	230	13

<sup>a</sup> ½-in. rollers except for 1½ in. O-rings where ¾-in. rollers were used.

<sup>b</sup> Castor oil used as lubricant between specimen and roller.

TABLE IV.—TENSILE STRENGTH AND ULTIMATE ELONGATION OF CUT RINGS.

Grip	Tensile Strength, psi				Ultimate Elongation, per cent			
	Clean Rollers		Castor Oil		Clean Rollers		Castor Oil	
	Mean	Limits	Mean	Limits	Mean	Limits	Mean	Limits
Single roller, rotated....	2270	100	2820	100	750	20	800	30
Single roller, free.....	830	60	2810	130	570	30	800	30
Double roller, ¾ in.....	1500	140	2900	100	680	60	810	30
Double roller, ½ in.....	1050	60	2930	100	620	40	800	30

used between specimen and grip. Some commercial products having a ring form apparently have sufficient mold release agent or bloom on the surface so that additional lubricant is not required. However, it is recommended that lubrication be required in standard methods of test since it is not known *a priori* whether sufficient lubricant is already on the specimen. On the other hand, standard method of test should permit freedom in choice of grips.

### Acknowledgments:

The single roller grips were supplied through the courtesy of Scott Testers, Inc. The O-rings were supplied through the courtesy of Goshen Rubber Co., E. F. Houghton and Co., Parker Appliance Co., and Precision Rubber Products Corp.

The assistance of George W. Bullman in making the tests is greatly appreciated.



# Tensile and Compressive Properties of Fiberglass Reinforced Laminates

By RICHARD E. CHAMBERS and FREDERICK J. McGARRY

A newly developed technique using integrally bonded foil resistance strain gages permits interior distortion measurements through the thickness of fiberglass-plastic laminates. This makes possible a detailed examination of orthotropic theory and an evaluation of the effects of certain parameter variations to better understand laminate behavior. Detailed studies of tensile, compressive, and flexural actions establish a consistent correlation between such actions and indicate that partial failure of the resin during tensile stressing takes place both in uniaxial and flexural tests. This failure, indicated by the knee in the stress-strain curve, has been further verified by moisture absorption under prolonged water immersion of laminate specimens before and after tensile stressing. Practical implications of this partial internal failure are noted.

## Experimental Procedure

The laminate studied was composed of 72 layers of Fiberglas cloth (Owens Corning No. 181-136) adhered with Paraplex P-43 polyester resin,<sup>2</sup> benzoyl peroxide catalyst, by the wet layup method. The laminate was press cured to  $\frac{3}{4}$  in. stops for 1 hr at 130 F and 6 hr at 150 F. During fabrication, electric foil strain gages, type FA-2,<sup>3</sup> were

ON THE BASIS of the mechanical properties of the components it is reasonable to expect that fiberglass-resin laminates may not retain their physical continuity except under unusual conditions of fiber orientation and modes of loading. This expectation arises from the differences in moduli of elasticity and coefficients of thermal expansion of the resin and the glass and from the low ductility of the resin prior to tensile or shear fracture (1).<sup>1</sup> Certain manifestations of the tendency of laminates to partially fail internally under stressing have been previously observed without specification in laboratory strength testing programs (2, 3) and in the performance of fiberglass reinforced plastic pipe (4). A more complete identification of the action, and its ramifications, has recently been obtained and constitutes the subject of this paper.

In the determination of the elastic moduli—tensile, compressive, and flexural—of a laminate two facts need be recognized:

1. The modulus values derived from tests on laminate specimens are strongly dependent upon the conditions of the test with respect to specimen size and geometry and the methods of parameter measurement (5), and

2. A consistent relationship between the modulus values should exist since flexural behavior involves both tensile and compressive stresses.

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<sup>1</sup> The boldface numbers in parentheses refer to the list of references appended to this paper.

<sup>2</sup> Rohm and Haas Co., Philadelphia, Pa.

<sup>3</sup> Baldwin-Lima-Hamilton Corp., Waltham, Mass.

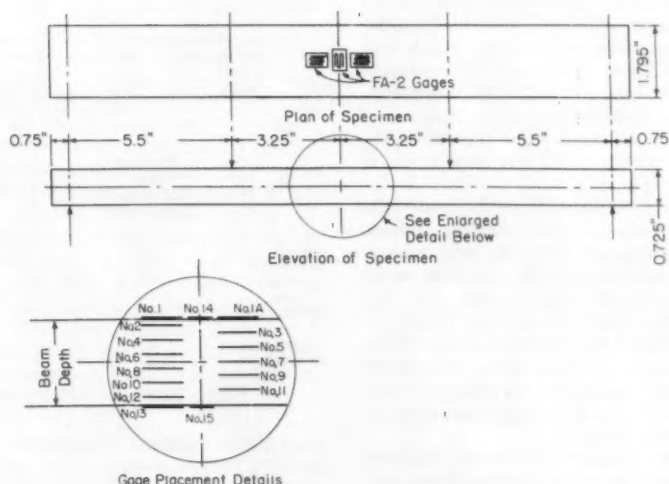


Fig. 1.—Bending specimen details showing location of strain gages.



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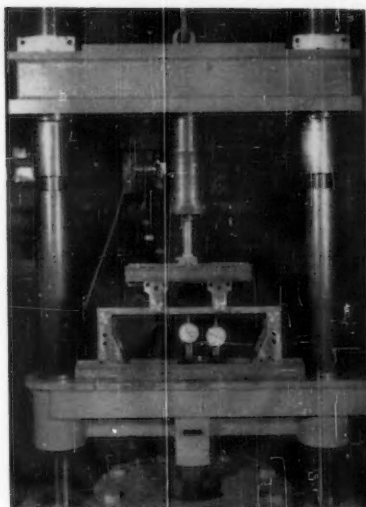


Fig. 2.—Bending jig details.

located at approximately  $\frac{1}{16}$ -in. intervals through the thickness of the laminate, in that portion of it destined to serve as a flexure specimen, as shown in Fig. 1. This technique of strain measurement, described in detail in previous publications (6, 7), makes possible a comprehensive study of the distortions undergone by a laminate specimen when subjected to various loads. The beam,  $\frac{3}{4}$  in. deep,  $1\frac{1}{2}$  in. wide, and 19 in. long, was loaded as shown in Fig. 2. Immediately adjacent to the flexure specimen in the plate, a tension specimen,  $\frac{3}{4}$  in. thick, 1 in. wide, and 17 in. long, and a compression specimen,  $\frac{3}{4}$  in. thick, 1 in. wide, and  $3\frac{1}{2}$  in. long, were taken from the laminate and fitted with longitudinal and transverse foil gages in pairs on opposite faces to compensate for stray bending effects.

A sequence of load-unload cycles was carried out on each of the three specimens to progressively higher, equal stress levels such that the stress history of the three specimens was common. This is described in Table I, which also includes the modulus data calculated for the various cycles and portions of cycles.

#### Test Results

Reference to the tension and compression data in Table I and the load-strain curves of Figs. 3 and 4 permit some interesting observations:

1. No changes in the compressive modulus occurred throughout the entire loading cycle;

2. In the first tensile loading to a particular stress level, an initial and secondary modulus phenomenon was apparent;

3. In the first tensile unloading from a particular stress level, the modulus was less than the initial but greater than

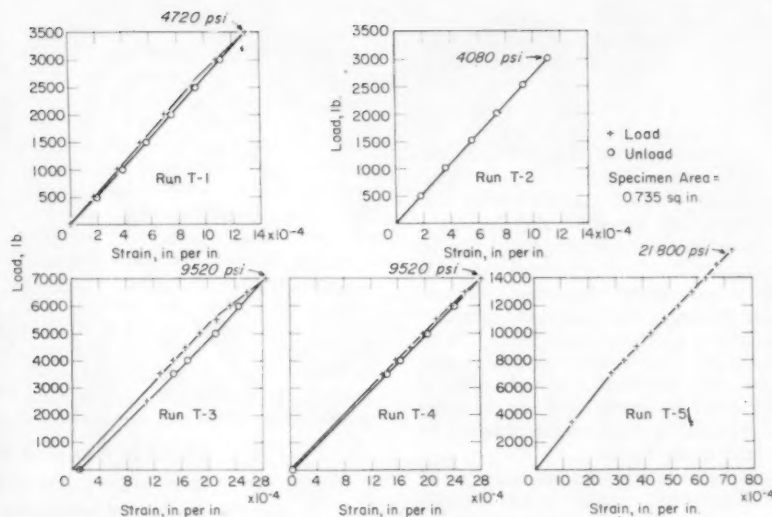


Fig. 3.—Plots of load versus strain for tension tests.

the secondary ones from the loading phase;

4. In the first tensile loading-unloading cycle an appreciable amount of hysteresis was apparent. This decreased or disappeared upon subsequent cycling;

5. In the second tensile loading to a particular stress level, the initial and secondary modulus differences were much smaller and closely approximated the value from the preceding unloading phase; and

6. As progressively higher tensile stresses were reached, the initial and

unloading moduli both continued to decrease and appeared to be approaching the value of the compressive modulus.

Most of these observations are corroborated by the data of Norris and Erickson (3) based upon a much more extensive test program.

The laminate flexural behavior is presented in Figs. 5 to 9. In Fig. 5(a), the action of each gage is linear as a function of load and the response of gage No. 7 indicates that the neutral axis coincides with the centerline of the beam, in accordance with assumptions of classical beam theory. Figure 6 pre-

TABLE I.

Run	Load, lb	Unload, lb	Stress, psi	Initial Modulus from Deflections, psi		
Flexure						
B-1.....	0 to 300	...	5 250	$3.25 \times 10^{-6}$		
B-2.....	0 to 600	600 to 0	10 500	3.30		
B-3.....	0 to 1100	...	19 300	3.31		
B-4.....	0 to 1100	1100 to 0	19 300	3.26		
B-5.....	0 to Failure	...	41 000	...		
Tension				Modulus, $10^6$ psi		
				Load		Unload
				Initial	Secondary	
T-1.....	0 to 3500	3500 to 0	4 720	3.84	3.16	3.70
T-2.....	0 to 3000	3000 to 0	4 080	3.67	3.67	3.67
T-3.....	0 to 7000	7000 to 0	9 520	3.62	2.59	3.44
T-4.....	0 to 7000	7000 to 0	9 520	3.44	3.40	3.43
T-5.....	0 to Failure	...	21 800	3.40	2.72	...
Compression				Modulus, $10^6$ psi		
				Load		Unload
				Initial	Secondary	
C-1.....	0 to 3 500	3500 to 0	4 600	3.37	3.37	3.37
C-2.....	0 to 3 500	3500 to 0	4 600	3.37	3.37	3.37
C-3.....	0 to 7 000	7000 to 0	9 200	3.38	3.38	3.38
C-4.....	0 to 7 000	7000 to 0	9 200	3.38	3.38	3.38
C-5.....	0 to 14 000	...	18 400	3.38	3.38	...

sents the strain and stress distributions through the depth of the beam occurring at approximately the maximum load in the first bending cycle. The stress distribution is obtained by converting the measured strain values to the appropriate stresses obtained from the previous uniaxial tests, tensile and compressive. Careful examination of Fig. 6 shows that the strain distribution is approximately linear until the two outer sixths of the beam thickness are reached; in these regions the strains become slightly larger than those indicated by a single straight-line distribution. With different tensile and compressive moduli and with the further complication of the secondary tensile modulus, the stress distribution is quite nonlinear as can be seen from the figure.

In Fig. 5(b), the influence of the initial and secondary tensile moduli becomes more apparent in the slight shift of the beam neutral axis, indicated by the tensile response of the centerline gage, No. 7, toward the compressive side of the beam. This occurs at a maximum fiber stress of about 7000 psi; approximately the same stress level at which the behavior became pronounced in tension. Some hysteresis, especially in the outermost gages, was apparent upon unloading, though the scale used in Fig. 5(b) does not well illustrate the effect. When the next higher stress level was reached, Fig. 5(c), the behavior was linear to approximately the previous load level, 700 lb, at which point a pronounced break in the load-strain responses of all gages took place with a further shift in the neutral axis. Since this action took place only in the tensile, uniaxial tests, it is apparent that the same phenomenon, partial tensile failure controls flexural behavior. As the stiffness of the tensile portion of the beam reduces, the neutral axis shifts toward the compressive side, exposing a greater volume of the beam to tensile strains and stresses. Again, as was the case in tension in runs T-3 and T-4, for example, a second application of the same flexural load, Fig. 5(d), shows a much more linear action with the accompanying flexural modulus reduction, Table I. The hypothesized internal failure thus appears to be irreversible and the hysteresis apparent in Fig. 5(d) indicates that one load-unload cycle accomplishes much but not all of the mechanical degradation.

Figure 5(e) shows the load-strain responses of the various gages as the beam was loaded to failure. Note the change in slopes as the previous load of 1100 to 1200 lb is passed and also the increasing neutral axis shift as more of the tensile fibers are subjected to higher stress and strain levels. Just prior to fracture, the strain distribution through

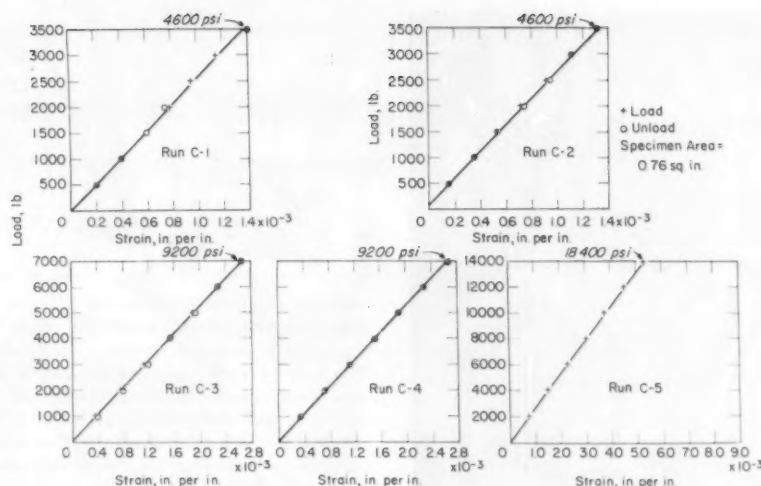


Fig. 4.—Plots of load versus strain for compression tests.

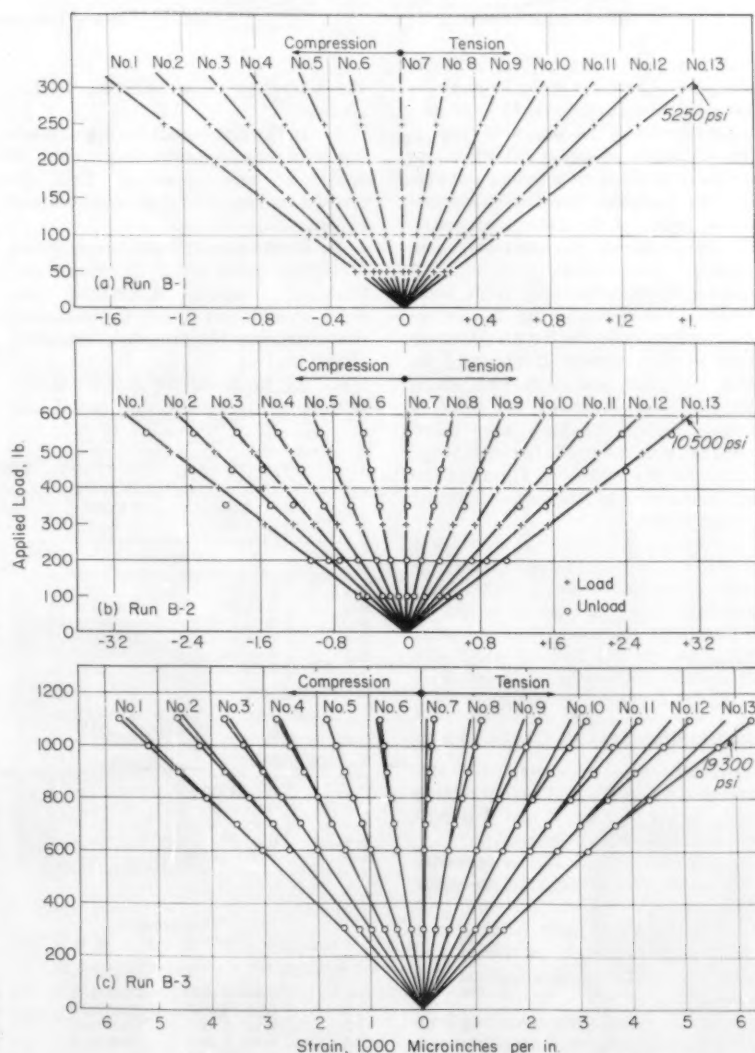


Fig. 5.—Plot of load versus strain for gages through the thickness of flexure specimen.



the depth of the beam was as shown in Fig. 7 where the neutral axis displacement is apparent and finite. That the shift was a progressive one is illustrated by Fig. 8 which shows the strain and stress distributions at one load level during run B-3. From Figs. 6 and 8, incidentally, it is possible to compute the resisting moments created by the forces resulting from the plotted stresses. Comparing these calculated moments to those resulting from the loads applied externally to the beam showed discrepancies of about 3 per cent—well within experimental error limits of the apparatus utilized.

To explore this curious tensile behavior of Fiberglass-resin laminates further, a second, simple experiment was performed. A 12-ply, 181 fabric P-43 resin laminate was fabricated in the fashion previously described and then cut into ten tension specimens,  $\frac{1}{8}$  in. thick, 1 in. wide, and approximately 8 in. long. Five of these, randomly selected, were loaded once in tension just beyond the first pronounced break or "knee" in the stress-strain curve and then unloaded. The remaining five specimens were never subjected to any loads. The ten were then machined to identical size and shape by cutting

off the ends, removing the grip-damaged areas from those which had been loaded. Next the ten blanks were immersed in a vessel of water at 72 F for a 34-day period during which time-weight measurements were frequently taken, as indicated in Fig. 9 and Table II, to observe water absorption. It is clear that those specimens which had been stressed in tension past the "knee" consistently absorbed more water, supporting the hypothesis that the internal failure so manifest actually consists of fractures on a very fine scale which probably take place in the resin or at the glass-resin interface. It has been previously pointed out that such could occur without complete destruction of the load-sustaining capacity of the structure (8).

### Conclusions

From the work described, the following conclusions appear valid:

1. In the tensile behavior of fiberglass-resin laminates, partial internal failures of the structure take place at stress levels low compared to the ultimate strength of the material and progress as the stress level under cyclic loading is increased.
2. Such failures do not appear to occur under compressive stressing.

3. The flexural behavior of fiberglass-resin laminates strongly reflects their tensile limitation and appears to be controlled by it.

4. At higher stress levels, the stress and strain distributions in flexural elements depart rather strongly from those classically predicted, and hence the evaluations of the flexural properties are in error, using the conventional simple formulas cited in most testing procedures.

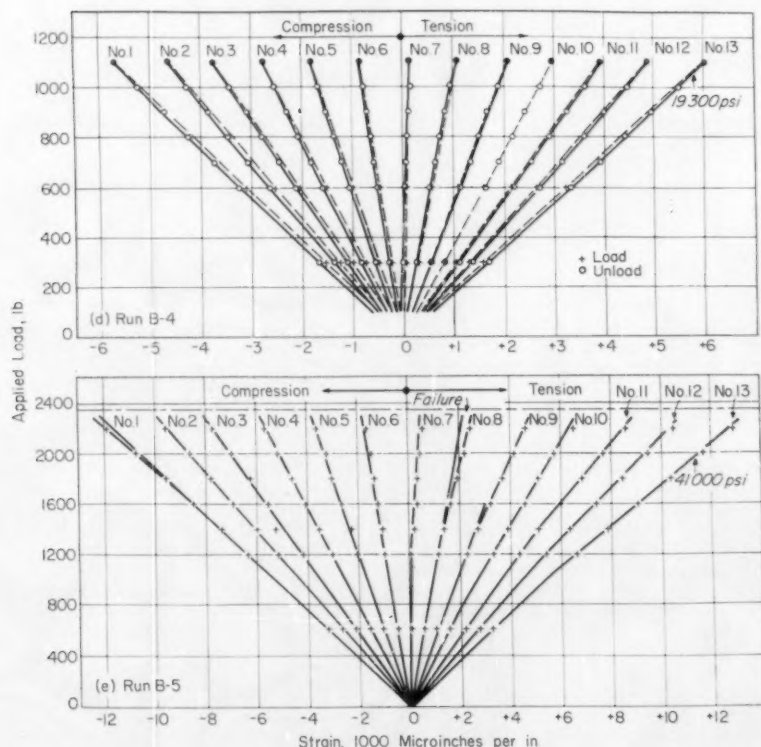


Fig. 5.—(Continued)

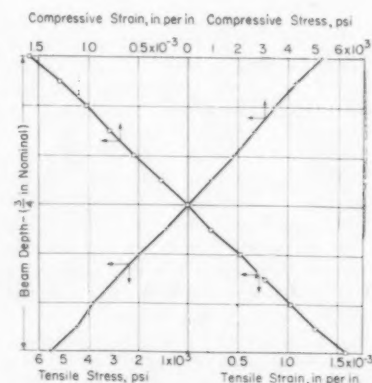


Fig. 6.—Stress and strain diagrams at an apparent fiber stress of 5360 psi, run B-1.

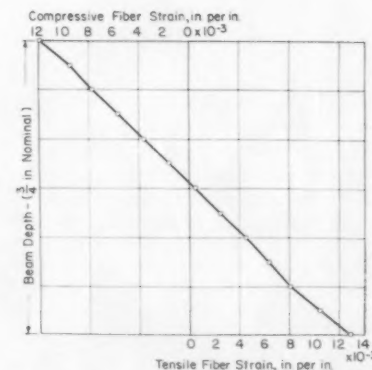


Fig. 7.—Strain distribution just prior to failure. (Apparent fiber stress of 36,000 psi), run B-5.

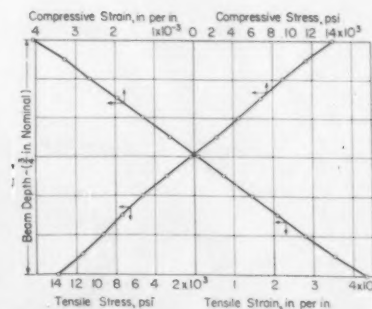


Fig. 8.—Stress and strain diagrams at an apparent fiber stress of 13,800 psi, run B-3.

5. Consistent with this, and previous, work and a consideration of the mechanical properties of the components, is the hypothesis that the internal failure mechanism under tension is a fracture failure.

6. The process of water immersion after stressing to various levels may be a profitable tool in laminate research, especially in the study of the effects of resin variables.

## Discussion

The investigation described suggests a number of interesting possibilities. Most prominent is the idea that the break in the tensile stress-strain curve indicates a maximum level to which a laminate can be stressed without degrading its structural integrity, maintenance of which is critically important for the performance of certain laminates exposed to deleterious media. It has been the experience of reinforced plastics pipe producers, for example, that the satisfactory working pressure must be much lower than the short-time bursting pressure of the pipe. Since the break in the stress-strain curve is usually 20 to 25 per cent of the static tensile strength of a laminate and good engineering judgment might choose a safety factor of two because of operating variables, this suggests a pipe working pressure of 10 to 12 per cent of its bursting pressure; a ratio empirically arrived at in the experience of several pipe fabricators.

In a recently published paper on fatigue study of fiberglass reinforced laminates, Boller (9) found that an approximate endurance limit at  $10^7$  cycles for polyester based laminates was 20 to 25 per cent of their static tensile strength. This form of behavior is extremely complicated but it would be interesting to see if a true endurance limit were exhibited by such materials at 5 to 10 per cent of their static strength.

As has been postulated previously (1) the role of resin ductility in the performance of laminates may be more critical than has been realized heretofore. Only under exceptional conditions of fiber orientation and load application do either epoxy or polyester laminates show the break in the tensile stress-strain curve at stress values higher than perhaps 30 per cent of their ultimate strengths. If careful strain measurements are made, hysteresis effects are apparent after cycling to even lower stress levels, suggesting that internal cracking commences very early in the life of the laminate. Greater resin ductility without a sacrifice in cohesive or adhesive strengths should operate beneficially in this respect.

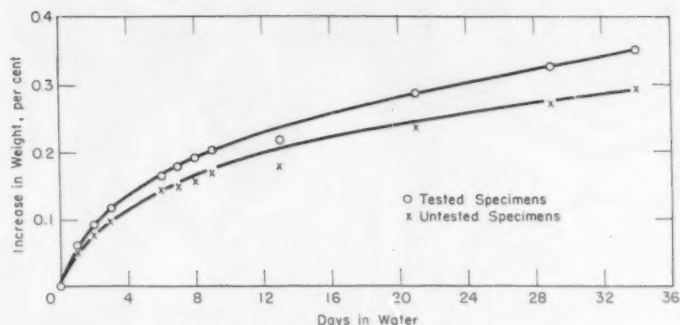


Fig. 9.—Water absorption of specimens showing effect of prior stressing.

TABLE II.

Specimen	Initial Weights Before Immersion, g	After 29 Days in Water at 70 F, g	Weight Change, g	Average Weight Change, g	Dry Weight, Moisture Pickup, per cent
No. 1.....	12.657	12.698	0.041	...	0.32
No. 2.....	12.672	12.713	0.041	...	0.32
No. 3.....	12.965	13.007	0.042	0.041	0.32
No. 4.....	12.445	12.486	0.041	...	0.33
No. 5.....	12.300	12.340	0.040	...	0.32
No. 6.....	13.194	13.230	0.036	...	0.27
No. 7.....	12.479	12.514	0.035	...	0.28
No. 8.....	12.824	12.858	0.034	0.034	0.26
No. 9.....	12.118	12.150	0.032	...	0.26
No. 10.....	12.400	12.432	0.032	...	0.26

## Acknowledgment:

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Gratitude is expressed to the following industrial associates who have contributed generously of their advice, assistance, materials, and experience: John H. Gibbud and William H. Otto of the Owens-Corning Fiberglass Corp. Arthur L. Smith and William G. Carson of the Rohm & Haas Co.; James E. Carey and Harvey L. Parry of the Shell Chemical Corp.

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# Beam and Column Tests of Welded Steel Tubing with Design Recommendations

By DON S. WOLFORD and M. J. REBHOLZ

Beam and column tests were performed on  $\frac{3}{8}$  to 3-in. diameter tubing made by forming and welding Armco Zincgrip flat-rolled steel of sheet gages from 20 to 12. Both low and medium carbon tubing was studied. Yield strengths in tubing were higher than the flat-rolled steel stocks from which they were made. Design yield strengths of 45,000 and 55,000 psi may be safely assumed for low and medium carbon tubing provided diameter-thickness ratios do not exceed 60 and 50, respectively.

Allowable design stresses of 27,000 and 33,000 psi are suggested for bending which insures a safety factor of 1.67 against design yield strengths and 30 to 50 per cent additional margin against maximum bending strength.

Combination straight-line Euler-type column formulas are recommended for welded steel tubing. These formulas take advantage of tubing's greater strength in the short-column range yet provide for recognized safety and eccentricity ratios.

Beam and column tests made at the same time on three small sizes of standard galvanized steel pipe show it may be adequately designed using stresses and formulas recommended by The American Institute of Steel Construction (AISC).

**W**ELDED carbon steel tubing is a product of flat-rolled steel, the latter having been reduced to desired gage then slit into narrow strips of proper width for the tubing size required. The slit strip is fed into a tube mill from a coil and continuously formed into round tubing complete with a straight resistance-welded seam. The weld flash is then trimmed before the tubing passes through the final roll stands which impart roundness, exact size, and smoothness (1).<sup>1</sup>

The structural advantages of round tubing are well known. It offers the ideal disposition of material to resist torsion. Columns and beams of round tubing are equally strong in all directions.

Design data available on tubing have largely been borrowed from procedures used for other steel structures, which do not utilize its strength to the fullest. In particular, the extra strength from cold forming into tubing has been ignored. This paper highlights the structural possibilities of such tubing for use in beams and columns of efficient yet safe design. Torsion properties have been investigated in the past (2).

## Specimens

Tubing made of both low (0.02 to

0.06 per cent) and medium (0.21 to 0.29 per cent) carbon Armco Zincgrip steels was investigated. These low- and medium-carbon compositions are similar to 1008 and 1025 steels and when supplied as galvanized strip in sheet gages have yield strengths of 35,000 to 40,000 and 40,000 to 45,000 psi, respectively. It should be noted that all tubing tested was primarily formed rather than drawn to size. Outside diameters of tubing investigated ranged from  $\frac{3}{8}$  to 3 in. with wall thicknesses from 0.038 to 0.112 in. All tubing used in testing was taken at random from production orders of Armco's Piqua, Ohio, tubing plant. Standard galvanized steel pipe was also tested for comparison in the three following nominal sizes:  $\frac{3}{4}$ , 1 $\frac{1}{4}$ , and 2 in.

Some of the tubing and pipe specimens used for compression property tests are shown in Fig. 1.



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## Mechanical Tests

Compression specimens were cut off in lengths three times the outside diameter of the tubing or pipe. Opposite ends of the specimen were made parallel within 0.001 in. in a lathe operation which also obtained flatness to the same degree of accuracy.

A spherical-seated compression head was used in a 200,000-lb Baldwin-Southwark testing machine to establish even loading. Three SR-4 type A-3 strain gages were mounted at 120 deg around the specimen at its mid-length and parallel to its axis. Load-strain pairs were observed "on the fly" at regular load intervals as the specimen was slowly compressed. A head speed of 0.005 in. per min was maintained until the strain in the specimen reached 0.005 in. per in.

The 0.2 per cent offset compression yield strengths ranged from 42,000 to 74,200 psi for low-carbon tubing, 50,900 to 66,700 for medium-carbon tubing and 33,800 to 41,000 for pipe (see Table I). Moduli of elasticity obtained in the compression tests ranged from 27,600,000 to 30,300,000 psi for tubing and 28,500,000 to 30,300,000 for pipe.

Tension tests were made according to ASTM Methods E 8-57 T,<sup>2</sup> Figs. 11, 12, and 6. For tubing 1 in. and smaller in outside diameter, the entire cross-section was tested. Specimens with 2-in. long by  $\frac{1}{2}$  in. wide gage dimensions were machined from tubing 1 in. in outside diameter and larger.



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**NOTE.**—DISCUSSION OF THIS PAPER IS INVITED, either for publication or for the attention of the authors. Address all communications to ASTM Headquarters, 1916 Race St., Philadelphia 3, Pa.

<sup>1</sup> The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 000.

<sup>2</sup> Tentative Methods of Tension Testing of Metallic Materials (E 8-57 T), 1957 Supplement to Book of ASTM Standards, Part 1, p. 437.



Tension specimens were pulled in a 20,000-lb Amsler testing machine. Load-deformation curves were obtained using a special 250X Microformer extensometer, built in the Armeo Research Laboratories, together with a Peters Microformer recorder. The 0.2 per cent offset tensile yield strengths obtained ranged from 42,000 to 73,600 psi in low-carbon tubing, 58,200 to 75,800 for medium-carbon tubing and 37,000 to 42,200 for pipe. Tensile strengths ranged from 56,100 to 75,000 psi for low-carbon tubing, 73,800 to 89,500 for medium-carbon tubing and 58,800 to 65,000 for pipe.

Tensile and compression yield strengths observed in each size of pipe were approximately equal in  $\frac{3}{4}$  and  $1\frac{1}{2}$ -in. sizes but tensile strengths exceeded compressive strengths by about 25 per cent in 2-in. size.

There was a tendency for tensile yield strengths to run somewhat higher than compressive strengths in tubing. Higher differences in yield strengths were observed in medium-carbon tubing than in low.

All reported strengths, both pipe and tubing, were slightly low for the steel since coated cross-sectional areas were used in reducing test loads to stresses. The same procedure was also followed in the structural tests, that is, coated thicknesses were used.

The first step in setting up design procedures is in establishing the lowest yield strengths that are likely to occur. Examination of yield strengths in Table I reveals that values of 45,000 and 55,000 psi can be used as minimum which are likely to occur in low and medium carbon tubing provided diameter-thickness ratios of 60 and 50 are not exceeded. Most of the values in each class are well above the minimums chosen. However, 3-in. diameter 18-gage tubing in both classes fell substantially below the minimums indicated so that ratios of diameters to thickness,  $D/t$ , values not to be exceeded have been stipulated. This limits use of the suggested minimum yield strengths to tubing in which the degree for forming is sufficient to raise yield strengths to the minimums indicated.

#### Beam Tests

Structural tests were limited to beams and columns. Application of these tests to design originated from the fact that they were made under controlled laboratory conditions convertible to practical applications.

The beam test shown in Fig. 2 is a method of applying third-point loading. At each load and reaction  $\frac{3}{8}$ -in. diameter rollers were used to permit large degrees of bending without excessive friction. The least ratio ob-

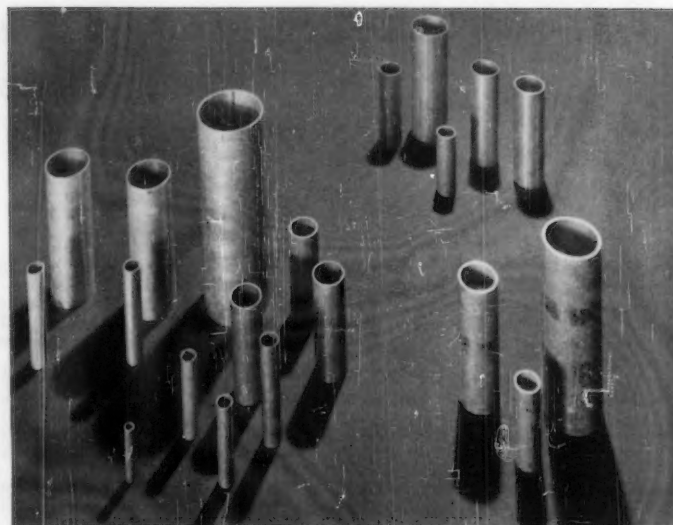


Fig. 1. Typical compression specimens.

Low carbon tubing at left with medium carbon at upper right and pipe at lower right.

served for radius of curvature to tubing diameter in the tests was approximately 30.

Test spans were made approximately 36 times the outside diameter of the specimen. This choice was influenced by a desire to conform with ratios of span length to diameter  $L/D$ , prevalent

in many tubing applications. Uniform curvature developed between the loads even at extreme degrees of bending until crippling occurred. Relatively thick tubing was as likely to cripple along the constant moment between loads as at the loads. Tubing having diameter-thickness ratios greater than

TABLE I.—MECHANICAL PROPERTIES—AVERAGES FOR DUPLICATE SPECIMENS OF TUBING AND PIPE.

Diameter, in.	Gage	0.2 per cent Offset Yield Strength		Tensile Strength, psi	Elonga- tion in 2 in., per cent	Com- pression Modulus of Elastic- ity, psi	Yield Strength in Com- pression versus Yield Strength in Tension	Diameter versus Thickness
		Tension, psi	Com- pression, psi					
Outside	Low Carbon Welded Steel Tubing							
$\frac{3}{8}$ ...	20	73 600	74 200	75 000	6	27.6 $\times 10^6$	1.01	8.5
$\frac{1}{2}$ ...	20	61 000	59 300	62 600	17	30.0	0.98	14.8
$\frac{5}{8}$ ...	18	65 400	62 200	67 700	16	30.3	0.95	11.4
$\frac{3}{4}$ ...	20	61 900	60 500	63 200	23	27.6	0.98	17.5
$\frac{7}{8}$ ...	18	57 600	59 800	64 100	21	28.7	1.04	13.2
$\frac{1}{2}$ ...	16	60 300	56 800	62 800	19	28.7	0.94	10.6
$1\frac{1}{8}$ ...	16	53 200	55 000	61 800	18	29.6	1.03	19.5
$1\frac{1}{2}$ ...	16	52 600	52 200	60 600	13	28.4	0.99	19.5
$1\frac{3}{4}$ ...	14	61 100	54 900	67 800	16	28.4	0.90	14.6
$2$ ...	18	49 300	50 000	60 700	22	28.7	1.02	25.8
$2\frac{1}{2}$ ...	18	51 000	51 700	60 000	20	28.7	1.02	36.8
$3$ ...	16	48 800	50 600	59 400	23	29.5	1.04	30.1
$3\frac{1}{2}$ ...	18	42 000	42 000	56 100	20	29.2	1.00	60.3
$4$ ...	14	45 600	44 400	56 600	24	29.8	0.97	37.3
Outside	C. MEDIUM CARBON WELDED STEEL TUBING							
$1\frac{1}{2}$ ...	14	75 200	66 700	89 500	13	29.6	0.89	10.3
$1\frac{3}{4}$ ...	16	75 800	61 800	87 400	14	29.0	0.82	19.1
$2$ ...	18	68 300	58 200	83 200	17	29.6	0.85	28.2
$2\frac{1}{2}$ ...	12	69 200	62 900	83 900	14	29.7	0.91	13.7
$3$ ...	20	71 800	54 800	81 800	18	29.3	0.76	50.5
$3\frac{1}{2}$ ...	13	58 200	57 300	76 600	18	29.3	0.99	25.6
$4$ ...	18	58 600	50 900	73 800	18	28.3	0.87	60.3
Inside	STANDARD GALVANIZED PIPE							
$\frac{3}{8}$ ...	..	39 300 <sup>a</sup>	41 000 <sup>a</sup>	63 600	26	30.3	1.04	8.2
$\frac{1}{2}$ ...	..	37 000 <sup>a</sup>	35 700 <sup>a</sup>	58 800	26	29.0	0.96	11.8
$2$ ...	..	42 200 <sup>a</sup>	33 800 <sup>a</sup>	65 000	29	28.5	0.80	14.8

<sup>a</sup> Yield point, lower in case of tension.

60 and 50 for low- and medium-carbon grades tended to fail at loads first unless reinforced. This was done by halving pieces of the same size of tubing, and twice its diameter in length, for insertion at loads and reactions. This procedure was not found necessary in testing pipe.

A unique feature of the beam test consisted of tracing the load-deflection curve on a chart as load was slowly increased. (A head speed of 0.05 in. per min was used.) A Leeds & Northrup two-coordinate drum-and-pen type recorder was fed voltages proportional to loads and deflections. A 6-in. long linear potentiometer bearing against the specimen at mid-span transduced deflections into proportional pen movements. Another linear potentiometer actuated from the pointer mechanism of the 200,000-lb Baldwin-Southwark testing machine caused the drum to rotate. Figure 3 shows the load-deflection curve obtained for a 1-in. diameter 14-gage medium-carbon tubing specimen tested on a 36-in. span. It is typical of all specimens tested.

Slope, a measure of flexural rigidity, and maximum load, a measure of ultimate bending moment, are the two curve quantities of most interest (see Table II). The stiffness ratio is the quotient of test and calculated flexural rigidities. The calculated flexural rigidity resulted from multiplying moment of inertia for measured cross-section by an assumed modulus of elasticity equal to 29,500,000 psi. The ratio ranged from 0.94 to 1.08 for tubing and from 0.98 to 1.03 for pipe, bearing out the reliability of the computed values.

Form factor is the quotient of test and calculated maximum bending moments. The calculated bending moments used resulted from multiplying section modulus based on measured cross-section by the average value of tensile and compressive yield strengths. The fact that form factors exceeding 1 and up to as high as 1.52 were observed showed that all specimens were sufficiently stable for well-advanced plastic straining to occur deep within the tubing under extreme bending. The  $\frac{3}{4}$ -in.

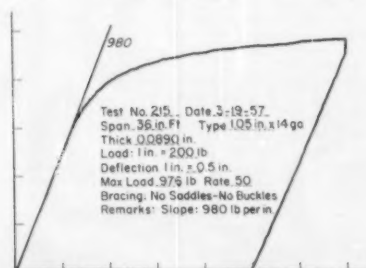


Fig. 3.—Typical load-deflection curve.

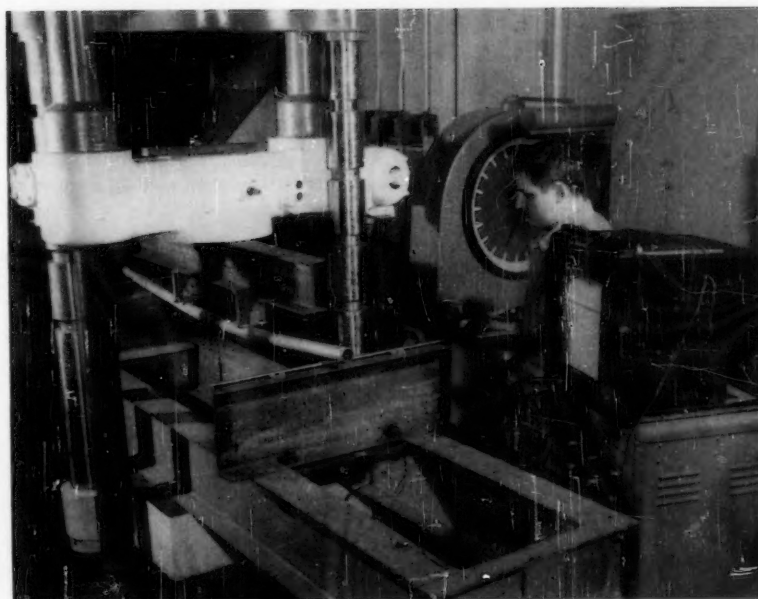


Fig. 2.—Beam test.

pipe showed a form factor of 1.60.

Another way to understand better the bending moment magnitudes observed is from apparent extreme fiber stresses obtained by dividing section modulus based on measured cross-section into maximum bending moment

by test (see Table II). The apparent extreme fiber stresses equaled or exceeded tensile strengths in all cases except for the 3-in. diameter 18-gage tubing, which was unstable, and in the  $1\frac{1}{4}$  and 2-in. pipe. The fault is that use of ordinary section modulus ignores

TABLE II.—BEAM TEST RESULTS—AVERAGES FOR DUPLICATE SPECIMENS OF TUBING AND PIPE.

Diameter, in.	Gage	Span <sup>a</sup> , in.	Flexural Rigidity by Test, lb-in./sq.	Stiffness Ratio <sup>b</sup>	Maximum Bending Moment by Test, in-lb	Form Factor <sup>c</sup>	Apparent Maximum Extreme Fiber Stress, psi
OUTSIDE							
LOW CARBON WELDED STEEL TUBING							
$\frac{3}{8}$ .....	20	18	17.8 $\times 10^3$	1.04	333	1.45	107 000
$\frac{1}{2}$ .....	20	24	28.2	1.00	804	1.38	82 900
$\frac{5}{8}$ .....	18	24	36.4	1.03	1 084	1.42	90 300
$\frac{3}{4}$ .....	20	30	172	1.03	1 315	1.42	87 500
$\frac{7}{8}$ .....	18	30	217	1.04	1 565	1.43	83 700
$1\frac{1}{8}$ .....	16	30	264	1.06	1 925	1.47	86 300
$1\frac{1}{2}$ .....	16	48	1 540	1.05	6 064	1.49	80 600
$1\frac{3}{4}$ .....	16	48	1 490	1.03	5 380	1.37	71 800
$1\frac{7}{8}$ .....	14	48	1 710	0.97	7 424	1.41	81 900
$2\frac{1}{8}$ .....	18	48	1 460	1.03	4 976	1.45	71 900
2.....	18	72	4 600	1.03	10 944	1.42	72 700
2.....	16	72	5 830	1.08	11 964	1.31	65 100
3.....	18	108	14 550	0.99	18 230	1.31	54 600
3.....	14	108	24 400	1.06	31 770	1.36	61 300
OUTSIDE							
MEDIUM CARBON WELDED STEEL TUBING							
1.....	14	36	795	0.98	5 856	1.52	107 800
$1\frac{1}{4}$ .....	16	48	1 230	0.97	6 960	1.48	101 900
$1\frac{1}{2}$ .....	18	54	1 710	0.94	6 795	1.32	83 200
$1\frac{3}{4}$ .....	12	60	4 430	0.99	18 000	1.33	96 300
2.....	20	72	3 410	0.98	9 852	1.32	83 500
$2\frac{1}{2}$ .....	13	84	12 600	1.02	30 100	1.47	85 000
3.....	18	108	14 440	1.01	22 400	1.29	69 100
INSIDE							
STANDARD GALVANIZED PIPE							
$\frac{3}{4}$ .....	..	36	1 090	0.98	4 560	1.60	64 200
$1\frac{1}{4}$ .....	..	60	5 600	1.01	12 200	1.45	54 000
2.....	..	84	19 800	1.03	25 830	1.20	47 300

<sup>a</sup> Spans were made at least 36 times outside diameter of specimen.

<sup>b</sup> Flexural rigidity by test divided by calculated flexural rigidity.

<sup>c</sup> Maximum bending moment by test divided by calculated maximum bending moment at yield strength.



Fig. 4.—Column test.

the nonlinear stress-fiber distance relations at high degree of bending, which in tubing is further confused by lack of a sharp yield point. What is significant is that an additional margin of safety is hereby provided, although no use has been made of it in the present work. Examination of load-deflection curves showed proportionality even above the calculated yield loads. This fact makes use of assumed minimum yield strengths from tension and compression tests entirely safe for bending design.

#### Column Tests

The 232 column tests made in a 200, 000-lb Baldwin-Southwark testing ma-

chine covered a range of slenderness ratios from 12 to 206, all with free ends. Eccentricity was kept within  $a = 0.10$ , except in one series of tests made on  $1\frac{5}{16}$ -in. diameter 16-gage tubing, for which a value of  $a = 0.25$  was used. The eccentricity factor,  $a$ , occurs in the secant column formula as follows:

$$f_y = \frac{P}{A} \left[ 1 + \frac{(ec/r^2) \sec(L/2r) \sqrt{P/AE}}{(1)} \right] \quad (1)$$

where:

- $f_y$  = yield point, psi,
- $e$  = eccentricity, in.,
- $c$  = extreme fiber distance, in.,
- $r$  = radius of gyration, in.,
- $L$  = unbraced column length, in.,
- $P$  = critical load, lb,
- $A$  = cross-sectional area, sq in.,
- $E$  = modulus of elasticity, psi, and
- $ec/r^2 = a$ , eccentricity factor.

Figure 4 shows a column being prepared for testing. Each end fitting consisted of two nested gimbals mounted at 90 deg to each other in four  $2\frac{1}{2}$ -in. diameter by  $1\frac{1}{2}$ -in. wide roller bearings. Column ends rested against a hardened steel plate, the face of which was located in the plane of the bearing axes. This eliminated the necessity for column-length corrections. Furthermore, there was little rotational constraint possible, even from lateral misalignment, however slight, since the end fittings were free to pivot in all directions.

Each end of column specimens had

been lathe-turned flat and normal to the specimen's axis. Any lack of straightness of the column specimen could have resulted in slightly nonparallel end faces, although end-fitting pivot action cancelled any restrictive tendency. Lack of straightness in a specimen was considered the same as end eccentricity in its effect on column load and was reckoned with accordingly in the analysis of results.

Eccentricity at ends of column specimens was purposely kept below 0.005 in. in all but the one series of tests on  $1\frac{5}{16}$ -in. diameter 16-gage low-carbon tubing which were offset in the same direction at both ends by 0.074 in. Ends of columns were positioned by means of calipers from four accurately located flats at 90 deg around the hardened seats.

Each specimen was checked against a straight edge before testing. Of the 232 specimens tested, 220 were straight within 0.010 in. per ft while 213 were straight within 0.005 in. per ft. No particular relation was found between weld position and crookedness.

Lateral deflections of column specimens adjusted to minimum end eccentricity were not measured. Deflections observable without instrumental aids were small until critical loads were approached. The series of eccentric tests of  $1\frac{5}{16}$ -in. diameter 16-gage low-carbon tubing ( $a = 0.25$ ) were made with a linear potentiometer resting lightly at column mid-length. Voltages produced by the deflections were fed

TABLE III.—COLUMN TEST LOADS—AVERAGES FOR DUPLICATE SPECIMENS OF TUBING AND PIPE.

Diameter, in.	Gage	Column Test Loads for Various Column Lengths, lb									
		12 in.	18 in.	24 in.	30 in.	36 in.	42 in.	48 in.	66 in.	84 in.	96 in.
OUTSIDE		LOW-CARBON WELDED STEEL TUBING									
$\frac{3}{8}$ .....	20	1 010	478	287	...	...	...	...	...	...	...
$\frac{3}{8}$ .....	20	3 040	...	1 424	...	...	478	...	...	...	...
$\frac{3}{8}$ .....	18	4 280	...	1 665	...	...	558	...	...	...	...
$\frac{3}{4}$ .....	20	4 200	3 500	...	1 702	...	...	692	...	...	...
$\frac{3}{4}$ .....	18	5 850	4 240	...	2 225	...	...	895	...	...	...
$\frac{3}{4}$ .....	16	6 520	5 250	...	2 675	...	...	1 045	...	...	...
$1\frac{1}{4}$ .....	16	12 700	...	10 090	...	8 865	...	5 675	...	1 997	...
$1\frac{1}{4}$ .....	16	11 900	...	10 625	...	8 150	...	5 475	...	1 908	1 460
$1\frac{3}{4}$ .....	16	11 400	...	9 525	...	7 200	...	5 175	...	1 880	1 430
$1\frac{3}{4}$ .....	14	820	...	14 700	...	11 250	...	7 410	...	2 425	...
$1\frac{3}{8}$ .....	18	10 060	...	9 840	...	8 040	...	5 410	...	1 892	...
2 .....	18	15 940	...	15 650	...	14 150	...	12 250	...	6 225	...
2 .....	16	18 750	...	18 260	...	17 300	...	14 910	...	7 350	...
3 .....	18	19 300	...	18 450	...	17 850	...	16 825	...	...	11 100
3 .....	14	33 400	...	31 350	...	29 140	...	29 520	...	21 300	...
OUTSIDE		MEDIUM-CARBON WELDED STEEL TUBING									
1 .....	14	15 150	...	10 920	...	...	...	3 300	...	...	...
$1\frac{1}{4}$ .....	16	13 500	...	11 150	...	...	...	5 375	...	...	...
$1\frac{1}{4}$ .....	18	12 880	...	11 050	...	...	...	7 450	...	...	1 910
$1\frac{3}{8}$ .....	12	32 300	...	30 120	...	...	...	17 220	...	...	4 750
2 .....	20	13 720	...	12 400	...	...	...	8 910	...	...	3 575
$2\frac{3}{8}$ .....	13	41 350	...	35 425	...	32 400	...	27 125	...	14 500	12 750
3 .....	18	25 500	...	23 225	...	21 550	...	19 450	...	...	12 125
INSIDE		STANDARD GALVANIZED PIPE									
$\frac{3}{4}$ .....	...	12 190	...	10 440	...	...	5 888	...	2 275	...	...
$1\frac{1}{4}$ .....	...	25 020	...	26 550	...	...	20 420	...	11 540	...	5 425
2 .....	...	36 500	...	36 480	...	35 280	32 850	...	38 250	...	19 920

\* 0.25 for this series only.



to the pen of the two-coordinated recorder discussed previously, while the same load-actuating arrangement was used to rotate the drum.

Lengths of columns from 1 to 8 ft were tested. Those of high slenderness ratios,  $L/r$ , suffered little permanent curvature from bending under critical load, while those of low slenderness ratios were permanently bent, and consequently weaker on reloading.

#### Column Formulas

Each critical load listed in Table III was the average of values obtained on two separate specimens.

Pipe critical loads were divided by allowable loads computed using AISC column formulas to obtain safety factors plotted in Fig. 5 (3). The AISC column formulas which follow are specified for the design of hot-rolled structural members of steel conforming to ASTM Specification A 7.<sup>3</sup> The AISC Manual lists allowable loads for 3- to 12-in. diameter pipe columns from these formulas, and they also may be presumed suitable for smaller sizes of pipe.

For main and secondary members with slenderness ratios,  $L/r$ , not greater than 120,

$$F_a = 17,000 - 0.485 (L/r)^2 \dots (2)$$

where:

$F_a$  = average axial stress, psi,  
 $L$  = the unbraced length of column, in.,

<sup>3</sup> Tentative Specification for Steel for Bridges and Buildings (A 7-56 T), 1956 Supplement to Book of ASTM Standards, Part 1, p. 185.

<sup>4</sup> Tentative Specification for Flat Rolled Carbon Steel (A 245-57 T), 1957 Supplement to Book of ASTM Standards, Part 1, p. 161.

<sup>5</sup> Tentative Specification for Hot-Rolled Carbon-Steel Strip of Structural Quality (A 303-52 T), 1955 Book of ASTM Standards, Part 1, p. 569.

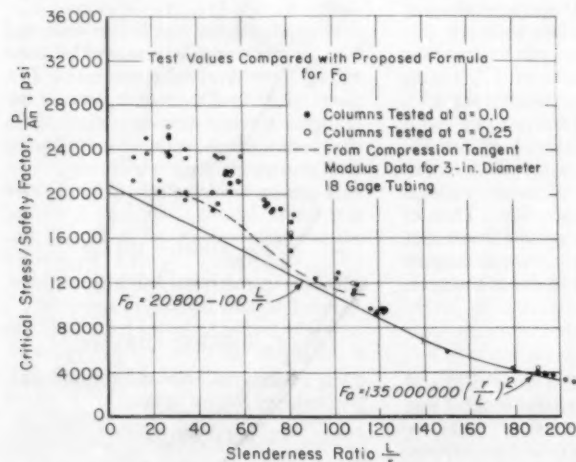


Fig. 6.—Critical stress to safety factor,  $P/A_n$ , versus slenderness ratio,  $L/r$ , for low-carbon welded Zincgrip steel tubing.

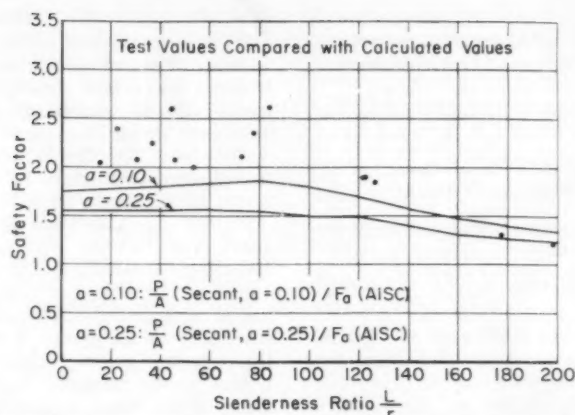


Fig. 5.—Safety factor versus slenderness ratio,  $L/r$  for standard galvanized steel pipe.

$r$  = corresponding radius of gyration of the section, in., and  
 $L/r$  = slenderness ratio.

For bracing and secondary members with slenderness ratios,  $L/r$ , between 120 and 200 (4),

$$F_a = \frac{18,000}{1 + L^2/18,000 r^2} \dots (3)$$

For main members with slenderness ratios,  $L/r$ , between 120 and 200 (5),

$$F_a = \frac{18,000}{1 + L^2/18,000 r^2} [1.6 - (L/200 r)] \dots (4)$$

Solution of the secant column Eq 1 with  $a = 0.10$  (tests) and  $a = 0.25$  (general conditions) (6) and  $f_y = 33,000$  psi, the minimum tolerable in a Specification A 7 steel,<sup>3</sup> gives maximum critical loads to be expected from which theoretical safety factors may be determined for the AISC formulas. Figure 5 shows such safety factors as solid lines. Experimental safety factors exceed theoretical safety factors in all but the longest columns. The comparison shows

that small sizes of pipe are amenable to design by means of the AISC column formulas with further refinement probably unwarranted.

Tubing demands special column formulas. The AISI Light Gage Cold-Formed Design Manual column formulas come near to meeting the need (7). However, the Manual is primarily intended for designing structural members from flat-rolled steel mechanical properties. Use of steels conforming to ASTM Specifications A 245,<sup>4</sup> and A 303<sup>5</sup> is stipulated with the strongest steel considered being grade C, for which yield strength must be at least 33,000 psi. Use of carbon steels of higher yield strength is not permitted unless a special procedure is followed, and even then it is intended for sharp-yielding steels rather than those made stronger by cold straining.

The AISI formulas follow:

For main and secondary members with slenderness ratios,  $L/r$ , equal to or less than  $24,000/\sqrt{f_y Q}$ ,  
 $F_a = 0.464 Q f_y - (2 Q f_y L / 100,000 r)^2 \dots (5)$

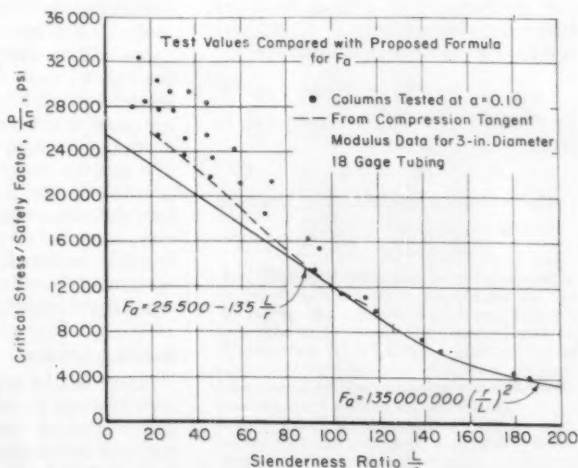


Fig. 7.—Critical stress to safety factor versus slenderness ratio for medium-carbon welded Zincgrip steel tubing.

in which  $Q = 1$  provided tubing ratios of diameter to thickness,  $D/t$ , do not exceed  $3,300,000/f_y$  and  $f_y$  = yield point of steel, psi.

For secondary members in which the slenderness ratio,  $L/r$ , is equal to or greater than  $24,000/\sqrt{f_y Q}$ ,

$$F_a = 134,000,000 (r/L)^2 \dots (6)$$

For main members with  $L/r$  ratios between 120 and 200,

$$F_a = 134,000,000 (r/L)^2 [1.6 - (L/200 r)] \dots (7)$$

Test critical loads were divided by allowable loads computed from the AISI formulas set up for yield strengths of 45,000 and 55,000 psi. These safety factors were plotted in the form of Fig. 5 and compared with theoretical safety factors obtained in the manner described previously for the AISC formulas. Critical loads for tubing were not quite high enough in the short column range to exceed consistently the safety factors demanded when the secant column formulas is used with  $a = 0.25$  to compute critical loads. This may be due to the nonlinear stress-strain relations of steel in tubing. The assumption of constant modulus of elasticity up to the critical load, upon which the AISI formulas are based, is not valid in this case.

It became apparent that a straight-line column formula comes nearer to fitting the short column data. Figures 6 and 7 each show a column curve based on the following formulas.

For low-carbon tubing (assumed minimum yield strength of 45,000 psi) and for  $L/r$  ratios from 0 to 139,

$$F_a = 20,800 - 100(L/r) \dots (8)$$

for  $L/r$  ratios from 139 to 200,

$$F_a = 135,000,000 (r/L)^2 \dots (9)$$

For medium-carbon tubing (assumed minimum yield strength of 55,000 psi) and for  $L/r$  ratios from 0 to 126,

$$F_a = 25,500 - 135 L/r \dots (10)$$

for slenderness ratios,  $L/r$ , from 126 to 200,

$$F_a = 135,000,000 (r/L)^2 \dots (11)$$

The preceding formulas stem from the following assumptions:

- basic safety factor of 1.73,
- possible eccentricity factor of  $a = 0.25$ ,
- $F_a$  at  $L/r = 0$  shall not exceed assumed minimum yield strength divided by  $1.73 (1 + 0.25) = 2.16$  (8),
- $F_a$  for long columns shall not exceed Euler value divided by 2.16, and
- $L$  shall be taken as the unsupported length of column, in.

Plotted values of Figs. 6 and 7 were obtained by dividing critical loads by

$n_1 = 1.73 (1 + 0.15) = 1.99$  rounded to 2.00, since eccentricity factor was  $a = 0.10$  (9). The series of 1 $\frac{5}{8}$ -in. diameter 16-gage low-carbon tubing columns tested with an eccentricity factor of 0.25 were divided by  $n_1 = 1.73 (1 + 0.0) = 1.73$ . This is proper since the effect of eccentricity has been reflected already in the critical loads.

Plotted allowable loads lie grouped above the formula curves. Group spreading was largely due to having in some specimens higher than the assumed minimum yield strengths. The space between the groups of points and the formula curves represents still further safety margin. Nothing was made of this since improving the fit would have entailed use of a cutoff.

The above column formulas are recommended for tubing used as both primary and secondary members. Equations 9 and 11 for long columns of tubing give average axial stress,  $F_a$ , values closely in line with those for the AISI formula 4 for pipe used as main members. The AISI Eq 7 cuts  $F_a$  still further in long columns. This may be desirable for open sections formed of flat-rolled steel in which twisting may cause failure at lower loads, but seems hardly applicable to tubing (10).

It was found that the relation between  $F_a$  and  $L/r$  could be fairly predicted from the compression stress-strain curves. Column curves derived directly from such data are plotted in Figs. 6 and 7. These were based on the weakest tubing of each carbon class judging by compression yield strengths. The tangent moduli of elasticity were determined at various stress levels. The column curve resulted from inserting pairs of stress and tangent modulus values in the Euler formula and solving for the slenderness ratio,  $L/r$ . The use of the Euler formula assumes no eccentricity, which was closely approached in the bulk of the column tests (11, 12).

A similar procedure was followed for the column tests made on 1 $\frac{5}{8}$  in. diameter 16-gage low carbon tubing with  $a = 0.25$  except that the secant column formula was used instead of Euler's. The column curve so obtained anticipated the critical load values by column tests but was on the low side. Thus, it may be stated that critical loads can be safely predicted by means of tangent modulus data obtained from compression tests of tubing.

## Bending Stresses

Safety factors implied from yield and allowable stresses stipulated in AISI and AISC manuals are  $33,000/18,000 = 1.83$  and  $33,000/20,000 = 1.65$ , respectively. In view of the extra margin of bending strength available in tubing

above yield strength value, a safety factor of 1.67 is suggested. It is close to the basic safety factor of 1.73 incorporated in the column formulas recommended herein for tubing.

The allowable stresses recommended for low and medium carbon tubing are then  $45,000/1.67 = 27,000$  and  $55,000/1.67 = 33,000$  psi, respectively.

## Summary and Conclusions

1. Welded carbon steel tubing in diameters from  $\frac{3}{8}$  to 3 in. and sheet gages from 20 to 12 both of low- and medium-carbon Armco Zingrip steel were tested as beams and columns along with  $\frac{3}{4}$ ,  $1\frac{1}{2}$  and 2-in. standard galvanized pipe.

2. Yield strengths found in welded steel tubing are higher than those found in the same steel before forming.

3. The relation between degree of forming expressed in the ratio of diameter to thickness,  $D/t$  and its effect on tubing strength is not precisely known. Design based on test data therefore seems logical.

4. Yield strengths of 45,000 and 55,000 psi may be assumed as minimums for design purposes in low and medium carbon tubing provided ratios of diameter to thickness,  $D/t$ , do not exceed 60 and 50, respectively. The recommended values embrace the lower yield strengths observed, whether from tension or compression tests.

5. The apparent extreme fiber stresses observed in welded steel tubing at high degrees of bending exceed yield strengths in tension or compression tests by 31 to 52 per cent, thus providing additional margin of safety.

6. Allowable stresses in bending of 27,000 and 33,000 psi are recommended for low- and medium-carbon tubing, respectively, and result from dividing assumed minimum yield strengths by 1.67.

7. Column tests with free ends and low eccentricity made on welded steel tubing over a slenderness ratio,  $L/r$ , range of 12 to 206 show it justifies use of higher average stresses particularly in the short column range. The following formulas are recommended:

Low carbon welded steel tubing,  $L/r = 0$  to 139,

$$F_a = 20,800 - 100 L/r$$

Medium carbon welded steel tubing,  $L/r = 0$  to 126,

$$F_a = 25,500 - 135 L/r$$

Long columns of low- and medium-carbon tubing,  $L/r$  up to 200,

$$F_a = 135,000,000 (r/L)^2$$

8. The column formulas given presuppose a minimum safety factor of 1.73

with an eccentricity factor of 0.25. They are therefore in line with customary practice and in the case of axially loaded straight columns result in safety factors as high as 2.16.

9. Column tests made on one size of tubing with an eccentricity factor of  $a = 0.25$ , consistent with the poorest acceptable practice, confirms the analysis used.

10. A column curve based on the Euler formula using pairs of stress and tangent modulus of elasticity values from compression tests in order to obtain slenderness ratio,  $L/r$ , values defines the lower bound of critical loads quite certainly.

11. Standard galvanized pipe of small sizes tested may be adequately designed using stresses and formulas recommended by AISC.

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# Effect of Calcination Temperature on Heat of Solution of Clays

By ALVARO LÓPEZ RUIZ

SINCE ancient times, it has been known that calcined clays can develop mechanical resistance if mixed with lime and water; that is, they obtain binding properties similar to those of the classic pozzolans. They are, in effect artificial pozzolans.

Pozzolans have recently been employed in the United States in the construction of dams because of the special properties of concrete manufactured with these materials (1, 2).<sup>1</sup>

The scarcity of natural pozzolan beds, their heterogeneity, and the presence of alkalis, may make it more practical to obtain pozzolans for the manufacture of pozzolanic cements by means of activation of clays which can be added to the portland cement, either at the cement plant or at the job site.

All clays are not, however, equally capable of being transformed into artificial pozzolans, nor are the best activation conditions always the same. The ability of each raw material to activate and the most favorable conditions for activation must be determined. The purpose of this paper is to establish a criterion for selection of the most suitable clays for transformation into artificial pozzolans and to establish a

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<sup>1</sup> The boldface numbers in parentheses refer to the list of references appended to this paper.

method to determine rapidly the optimum activation conditions.

Clay is a sedimentary deposit which consists principally of a complex mixture of characteristic argillaceous minerals. Clays are usually denoted by the name of the mineral which is present in greatest proportions—for example, kaolinitic, montmorillonitic, or illitic clays (3).

The crystalline structure of argillaceous minerals is fundamentally determined by the silicon, oxygen, aluminum, and hydroxyl ions. The clay water may be adsorbed, interlayer, or lattice water. When the material is heated it loses its water in that order, indicating the relative energy with which the water is bound to the crystallographic structure.

The interlayer water depends fundamentally upon the existence of exchangeable cations and negative charges in excess in the layers which integrate the argillaceous minerals. The charges in excess are due to atomic isomorphic substitutions—for example aluminum by iron or magnesium, and silicon by aluminum. The lattice water appears in the crystalline structure of the argillaceous minerals in the form of OH-ions.

The transformations which take place on heating the clay minerals may explain the mechanism of clay activation.

Figure 1 shows loss in weight of a kaolinite as a function of the temperature. In the figure, one can see that up to 400 C, there is only a slight loss of

weight; between 450 and 550 C, however, the dehydration curve experiences a sudden transition which corresponds to a loss of weight of nearly 13 per cent. Above this temperature, the loss is very small.

The differential thermal analysis of kaolin results in a diagram similar to Fig. 2. This figure shows an intense endothermic effect, the maximum of which is around 580 C, and a sudden exothermic effect around 950 C. Around 150 C there is also a slight endothermic effect. This is interpreted as follows:

Up to 400 C, kaolin loses its adsorbed



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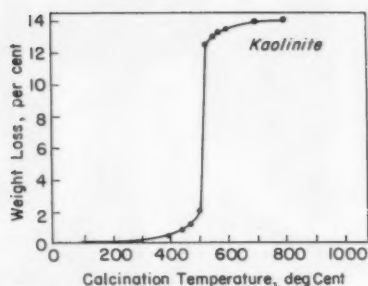


Fig. 1.—Dehydration of a Kaolinite by Calcination.

From Fig. 3, R. C. Mielenz, L. P. Witte, and O. J. Giantz, "Effect of Calcination on Natural Pozzolans," Symposium on Use of Pozzolanic Materials in Mortars and Concrete, Am. Soc. Testing Mats., p. 57 (1950). (Issued as separate publication ASTM STP No. 99.)

water (it has practically no interlayer water), giving rise to a small endothermic effect. Between 450 and 600 C there is a remarkable loss of weight resulting from the loss of lattice water formed from the OH-ions of the crystalline structure. At the same time the crystalline structure is destroyed and converted to an amorphous product (as has been shown by X-rays) probably formed by free silica and alumina. The sharp exothermic effect produced at temperatures near 950 C is believed to be due to the crystallization of  $\gamma$ -alumina from the amorphous alumina previously formed.

In montmorillonitic and illitic clays, endothermic effects appear near 600 to 700 C, as shown in Fig. 3. They are occasioned by loss of lattice water. The exothermic effect near 900 to 1000 C results from a crystallization process less marked in this case than in the kaolinitic clays. Both the montmorillonitic and illitic clays exhibit marked endothermic effects at low temperatures as a result of the expulsion of interlayer water. This is especially true in the case of montmorillonitic clays.

The amorphous product obtained in the endothermic process has a high energy level or chemical reactivity. Therefore, it appears desirable to obtain the greatest possible amount of amorphous silica and alumina with the maximum activity when obtaining artificial pozzolans from clays.

We may say, in principle, then, that in a clay of any specific mineralogic type, the more lattice water it possesses the more fit for activation it will be, that is to say, the more marked is the sudden transition observed in the loss of weight-temperature analysis or the greater the endothermic effect evidenced by the differential thermal analysis.

As a routine test for determining the lattice water of a clay, we propose the method which consists of deter-

mining the weight of the water emitted when a clay sample which has been previously desiccated at 300 C is calcined in a Penfield tube. This method avoids the possible errors caused by carbonates or other substances which when calcined emit vapors other than water.

The selection of 300 C as drying temperature for the determination of lattice water of clays is justified, in the most delicate case of montmorillonitic clays, by Ross and Hendricks (4) who determined that the lattice water lost at temperatures lower than 300 C is practically the same as the water lost at higher temperatures. With kaolinitic clays the temperature of 300 C presents no difficulty since there is no interlayer water and the loss of lattice water is accomplished at higher temperatures.

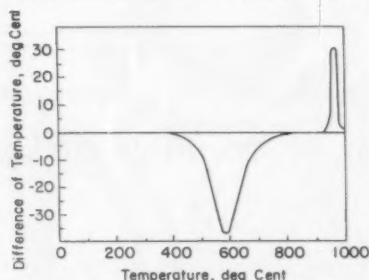


Fig. 2.—Differential Thermal Analysis of a Kaolinite.

The case of illitic clays, which generally have a smaller proportion of interlayer water than the montmorillonitic ones, may be considered intermediate between these and the kaolinitic clays. The author sees no reason for not accepting the 300 C temperature for previous desiccation of the illitic clays in the test considered here.

TABLE I.—LATTICE WATER OF SOME CLAYS AND STRENGTHS OF ACTIVATED CLAY AND LIME SPECIMENS.

	Lattice Water, per cent	Flexural Strength, psi	Compressive Strength, psi
Kaolinite A....	11.90	500	1780
Kaolinite B....	3.25	425	1180
Illite A.....	7.00	355	855
Illite B.....	5.00	255	655
Illite C.....	4.30	185	400

The possible presence in clays of aluminum or iron hydroxides which should lose their lattice water at temperatures higher than 300 C does not substantially diminish the value of the test, since these compounds also acquire pozzolanic activity by thermal treatment. This is particularly so in the case of aluminum hydroxides.

Table I shows the percentage of lattice water and the mechanical resistance of prismatic specimens of 2.5 by

2.5 by 10 cm which were manufactured with various kaolinitic and illitic clays activated under optimum conditions (previously determined) and mixed with calcium hydroxide in the proportion 3:1 (3 parts by weight of activated clay plus 1 part by weight of calcium hydroxide). The specimens were kept 7 days in the molds and 21 days in water prior to testing. Mixing water to obtain normal consistency was added.

The values shown in the table indicate a good relationship between the lattice water and the strengths of the activated clay-lime mixtures, in the same clay families.

From the table it is also concluded that the kaolins, even when having a smaller proportion of lattice water, give higher resistances than the illitic clays we have studied.

In order to choose the best temperature for activation of clays, we may well be guided by the results of the differential thermal analysis. The required temperature should be between that of the destruction of the crystalline structure and the crystallization of  $\gamma$ -alumina. The temperature interval, nevertheless, is too large (for instance, approximately 400 C in the kaolinites) so that the indications of the differential thermal analysis will not be immediately apparent.

Furthermore, in practice, there is another factor of fundamental importance from the point of view of the effective utilization of the process, that is, the length of the activation treatment of the clay, which should be minimum. Once the destruction of the crystalline structure has been accomplished, it is convenient to cool the product rapidly in order to "freeze" the structural state, especially if the material has been heated at maximum temperatures compatible with the activation process, with the object of accelerating it.

In order to determine the optimum conditions of activation it is necessary to use the strength test, at 28 days, of the specimens fabricated with activated clay and hydrated lime. This method is time consuming and requires large samples of the product. In lieu of this

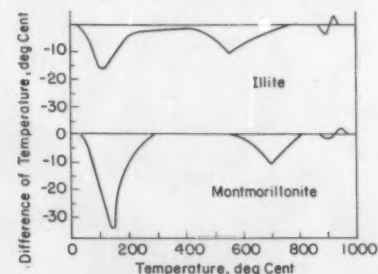


Fig. 3.—Differential Thermal Analysis of an Illite and Montmorillonite.

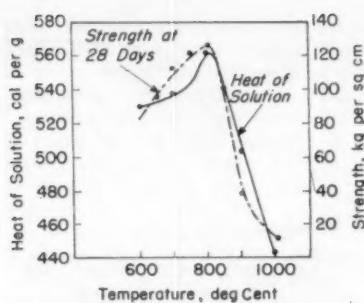


Fig. 4.—Heat of Solution of Kaolin and Compressive Strength of the Activated Kaolin-Lime Specimens, as a Function of the Activation Temperature.

method, the determination of silica and alumina soluble in acids has been proposed as a quality index. With this method, which is also somewhat lengthy, the author has not obtained good correlation of results and has considered the possibility of establishing a calorimetric method by which to determine the optimum activation conditions.

The proposed test is based on the hypothesis that the optimum activation conditions for a given material will be those which determine the maximum heat of solution of the product in an appropriate acid mixture. It is assumed that the more complete the dehydration obtained and the greater the proportion and activity of the amorphous phase produced, the greater the heat will be.

The test method is analogous to that used in the determination of the heat of hydration of a cement (5). The only difference is the use as the attacking liquid of 380 ml of 2N nitric acid and 20 ml of 40 per cent hydrofluoric for a sample of approximately 3 g. The determination takes approximately 1 hr.

One can calcine another sample simultaneously, which permits the determination of the amount of water retained by the activated clay. The heat of solution found is referred to that of the sample free of water.

With respect to pozzolanic cements (6), upon determining the heat of solution of activated clays with the acid solution prescribed in the British Standard: 12:1947 for portland cement, one does not normally obtain the complete solution of the sample. That is why the amount of hydrofluoric acid shown in the British standards was doubled. Even though in many cases one does not obtain total solution in a reasonable time, the reaction is nearly complete. We accordingly use this reagent and a contact time of 40 min for all tests.

Under these conditions all the heats of solutions were determined and were referred to the total of sample added to the calorimeter.

It is logical to suppose that the small amounts of sample which do not dissolve during the test are inert, that is, they lack pozzolanic properties. Therefore, although the heat of solution thus determined is, in some cases, lower than the true one, it may be adopted as an index of the activity that the clay has obtained with the thermal treatment.

In the following discussion, we present in detail experience with three kaolins, one montmorillonitic clay, and three illitic clays, which demonstrate the existing relationship between the activation conditions, the solution temperature, and the mechanical resistances of the activated clay-lime specimens.

#### Heats of Solution of Thermally Activated Kaolins and Mechanical Properties of Activated Kaolin-Lime Specimens

In order to study the variation of the heat of solution of kaolinitic clays with the activation temperature, three different samples of kaolin were tested: the first one of foreign origin and of a quality specified in the German pharmacopeia, the second rich in lattice water, and the third with little lattice water.

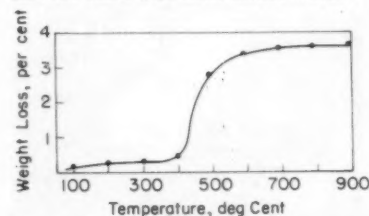


Fig. 5.—Loss of Weight versus Temperature Analysis of Kaolin Which Is Poor in Lattice Water.

#### Tests Made with Pure Kaolin

This material was activated by putting 10 g of powdered kaolin in an electric oven provided with an automatic temperature regulation device for a period of 1 hr at different temperatures. The product was rapidly cooled in the open air, on an iron plate. The results obtained are shown in detail in Table II.

From the values shown in Table II we deduce that by increasing the treatment temperature the heat of solution increases up to a maximum value, after

TABLE II.—HEAT OF SOLUTION OF A PURE KAOLIN HEATED FOR 1 HR AT DIFFERENT TEMPERATURES.

Temperature, deg Cent	Heat of Solution, cal per g
No activation	376
500	581
600	630
700	650
800	645
900	638
1000	494
1100	443

which it decreases.

From these values, bearing in mind the above-mentioned considerations, we may conclude that the best activation temperature under the prescribed conditions will be 700 to 750 C approximately.

Since the kaolin is a pure product and, therefore, without practical interest, the heat of solution at intermediate temperatures was not studied, nor were mechanical tests made.

#### Tests with Kaolin Rich in Lattice Water

The lattice water of the kaolin sample studied was 11.9 per cent.

The activation procedure of this kaolin was the same as that for the pure kaolin, except that the amount of the sample was 250 g, permitting the subsequent manufacture of activated specimens mixed with lime for strength tests.

The specimens were made by combining three parts activated kaolin and one part slaked lime by weight. Mixing water was added to obtain normal consistency. The specimens were broken at 28 days, after being kept for 7 days in the moisture room at 20 C and 21 days in water. Their dimensions were 2.5 by 2.5 by 10 cm, and they were broken first in flexure and then tested in compression.

Figure 4 shows the heats of solution and the compressive strengths of the specimens as a function of the activation temperature. The values shown correspond to the average of those found experimentally.

It is observed in Fig. 4 that both curves present a well-marked maximum at the activation temperature of 800 C. The average dry density of the activated kaolin-lime specimens was 0.9.

#### Tests with a Kaolin Low in Lattice Water

The tests made with the kaolin low in lattice water (3.25 per cent) were analogous to those carried out with the kaolin mentioned above.

The loss of weight temperature analysis is shown in Fig. 5. The heat of solution and the compressive strengths of the specimens fabricated with this kaolin are shown in Fig. 6, as a function of the activation temperature.

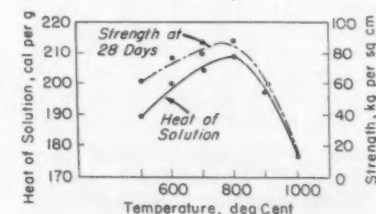


Fig. 6.—Heat of Solution of Kaolin and Compressive Strength of Activated Kaolin-Lime Specimens, as a Function of the Activation Temperature.

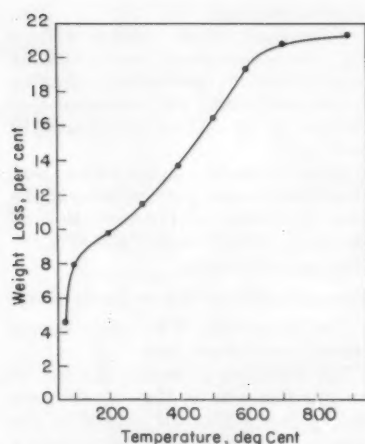


Fig. 7.—Loss of Weight versus Temperature Analysis of a Bentonite.

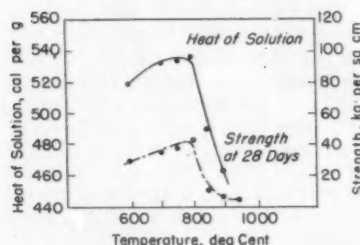


Fig. 8.—Solution Temperature Curves of a Bentonite and Compressive Strengths of the Activated Bentonite-Lime, as a Function of the Activation Temperature.

The test procedure is the same as for the other samples.

As in the previous case, both curves indicate a maximum for the same activation temperature—800 C. The maximum is less marked since both compressive strengths and heats of solution are definitely less than those indicated for the kaolin rich in lattice water.

#### Heats of Solution of Thermally Activated Bentonite and Strengths of Activated Bentonite-Lime Specimens

The investigation was continued by means of similar tests with a clay rich in minerals of the montmorillonite family, such as bentonite.

The loss of weight—temperature analysis of bentonite is given in Fig. 7. The lattice water of bentonite was found to be 10.1 per cent. The tests performed on the bentonite, under the same conditions as those for the kaolin, gave the results shown in Fig. 8.

#### Heats of Solution of Illitic Clays Thermally Activated and Strength of the Activated Clay-Lime Specimens

For the experiments with illitic clays, three clays used in the manufacture of ceramic bricks were tested.

The differential - thermal analysis shown in Fig. 9, of the three illitic clays, A, B, and C, confirms the characteristics in illitic clays.

The loss of weight - temperature analysis of clay A is shown in Fig. 10.

The lattice water contents of clays A, B, and C are given in Table III.

TABLE III.—LATTICE-WATER CONTENT OF CLAYS A, B, AND C.

	Lattice Water, per cent
Clay A.....	7.0
Clay B.....	5.0
Clay C.....	4.3

The activation experiments with illitic clays, A, B, and C, were performed in a manner similar to those described previously.

Figure 11 shows the heats of solution of clay A heated for 1 hr as a function of the activation temperature and the compressive strength of the specimens fabricated with the clay-lime mixture. The testing conditions are the same as for the other samples.

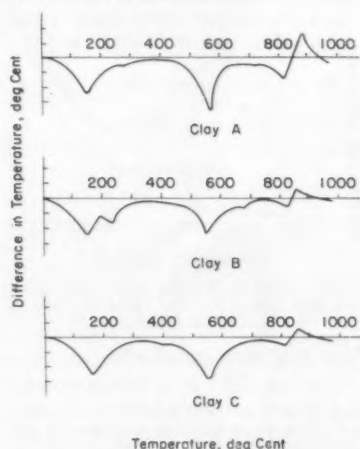


Fig. 9.—Differential Thermal Analysis of Clays A, B, and C.

It is noted that both curves have a maximum near 800 C.

Clay B was treated similarly for 1 hr in the electric oven at different temperatures; the heat of solution of the activated samples and the corresponding strengths are given in detail in Table IV.

TABLE IV.—HEAT OF SOLUTION AND STRENGTHS OF CLAY B, THERMALLY ACTIVATED. TIME OF ACTIVATION, 1 HR.

Activation Temperature, deg Cent	Heat of Solution, cal per g	Flexural Strength at 28 Days, psi	Compressive Strength at 28 Days, psi
650.....	358	228	570
700.....	368	228	597
750.....	380	256	654
800.....	360	185	555
850.....	352	100	256

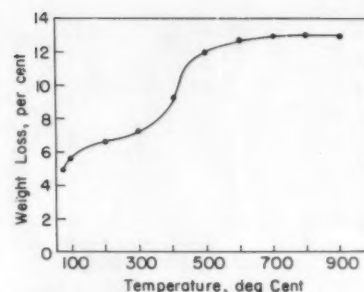


Fig. 10.—Loss of Weight versus Temperature Analysis of Clay A.

The maximum heat of solution and greatest strength is near 750 C. The activated sample at that temperature reached a compressive strength of 654 psi, which is in accordance with the lower percentage of lattice water.

Results for clay C, activated under conditions similar to those used for clays A and B, are shown in Table V.

TABLE V.—HEAT OF SOLUTION AND STRENGTHS OF CLAY C, THERMALLY ACTIVATED. TIME OF ACTIVATION, 1 HR.

Activation Temperature, deg Cent	Heat of Solution, cal per g	Compressive Strengths at 28 days, psi
650.....	338	270
700.....	346	370
750.....	349	398
800.....	342	356

It is again noted that the heat of solution is proportional to the strengths which were obtained with the activated clay-lime specimens; in other words, the heat of solution is an index of the pozzolanic activity obtained with the treatment given to a particular clay sample.

#### Influence of Temperature and Activation Time on the Heat of Solution and the Strengths of Activated Clay-Lime Specimens

In the industrial process of clay activation, it is desirable that the treatment time be held to a minimum. Therefore it is necessary to consider the influence of activation time, at different tempera-

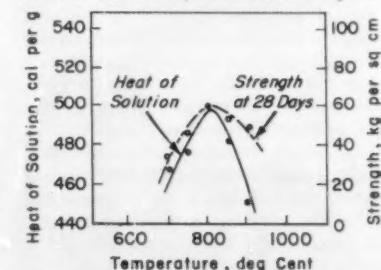


Fig. 11.—Heat of Solution of Clay A and Compressive Strength of the Activated Clay-Lime Specimens, as a Function of the Activation Temperature. Time of Treatment 1 hr.



tures, on the quality of the product. For this purpose, activations with an illitic clay at different temperatures and times were made, and the heats of solution of the activated samples were determined, as well as the strengths of a clay-lime mixture (in proportion of 3:1) at 28 days (7 days in the air and 21 in water).

The raw material was an illitic clay similar to clay A but from a different lot (called A'). The results obtained are given in detail in Fig. 12.

The tests performed with the bentonite, gave the results shown in Fig. 13.

As will be observed, both groups of experiments give similar results and permit the following conclusions:

1. The heat of solution curves for the activated clay and those of compressive strength for the specimens of activated clay lime show a correlation, but the pairs of curves corresponding to different activation temperatures have a different general form, depending on whether the activation temperature is above or below a specific temperature. This temperature is 800 C, for both samples tested: bentonite and illitic clay A.'

The curves of the activations realized at 850, 900, and 1000 C show a definite maximum which corresponds to the best activation time.

If the activation is at a temperature of 800 C, or lower, the maximum does not appear, but instead an asymptotic value results, not only for the heat of solution curve but also for the compressive strength as a function of time. This is explained by the fact that at 800 C, and at lower temperatures, the crystallization processes of the amorphous phase and the formation of new, practically inert, crystalline compounds do not take place, or if they do occur, they occur at a very slow rate. Above 800 C, these processes would take place with considerable speed, the higher the temperature the greater the speed. Thus, if the activation temperature is 800 C or less, the activity increases with the duration of treatment, until the completion of the dehydration and activation process; from this moment it remains practically constant. This explanation justifies, moreover, the similarity between the heat of solution and compressive strength curves of the activated clay-lime mixtures as a function of time, regardless of the activation temperature.

At 850, 900, and 1000 C, the clay dehydration would take place almost instantaneously. However, after a certain time, the crystallization processes or the formation of new compounds might become important, at which time the activity and heat of solution and the compressive strength of the activated

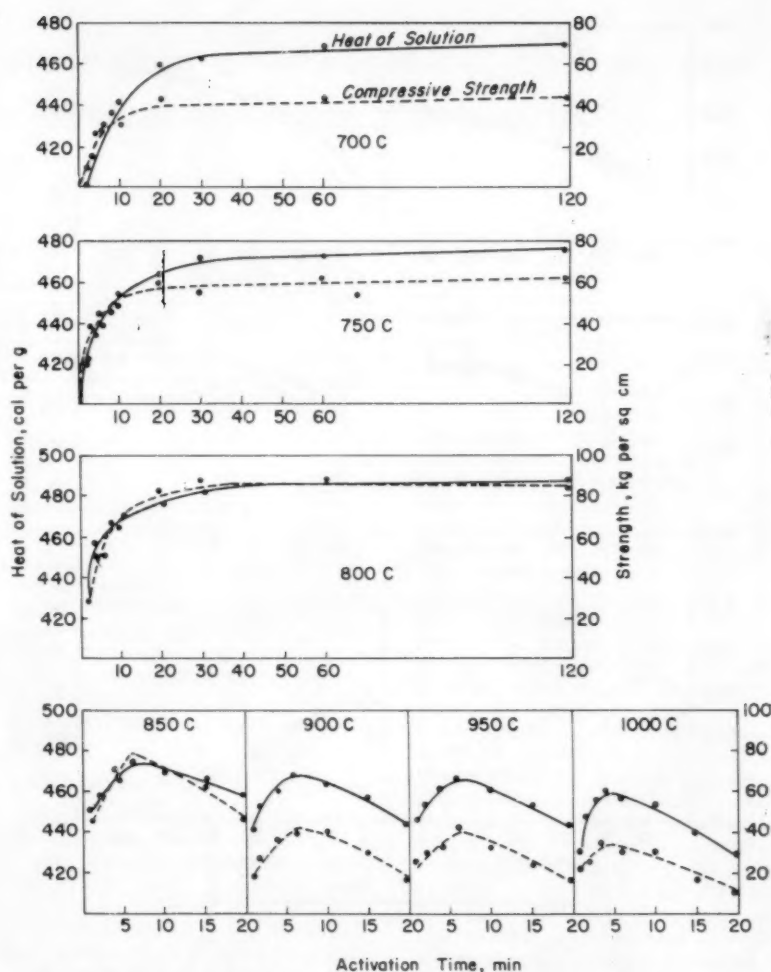


Fig. 12.—Heat of Solution of an Illitic Clay Activated at Different Temperatures and Times, and Compressive Strength of Activated Clay-Lime Specimens.

clay-lime mixtures should diminish perceptibly. In this way, the existence of a maximum is explained for the above curves, which show the best activation time for the temperature in question.

It is obvious that in industrial practice it is not advisable to exceed the maximum temperature which causes an activation curve with an asymptote, as a function of time, if one does not possess a kiln with good temperature control.

2. The maxima obtained in the last-mentioned curves are produced for periods which become shorter and shorter as the temperature increases and correspondingly diminish in value. This may be explained by assuming that, starting at 800 C even for shorter periods of time, simultaneous processes of crystallization or formation of new, practically inactive, compounds take place, and the higher the activation temperature, the greater the reaction will be, with increasing loss of the pozzolanic

power of the calcinated product.

3. The absolute value of the heat of solution of a specific clay, considered separately, may lead to error in some cases with regard to the activity of this activated clay. This is so, for example, if the activations were made with the samples tested above 800 C.

According to the shape of time-heat of solution curves, at different activation temperatures (Figs. 12 and 13), if a constant activation period of 1 hr is maintained and the activation is tested first at different temperatures, the resulting curve representing the heat of solution as a function of temperature presents a maximum which corresponds to the maximum temperature for which the heat curves of time-heat of solution continue to increase in an asymptotic way. If the activation is tested later at this temperature, for variable periods, it is easy to fix the optimum duration for the activation treatment, which will correspond to the highest compressive

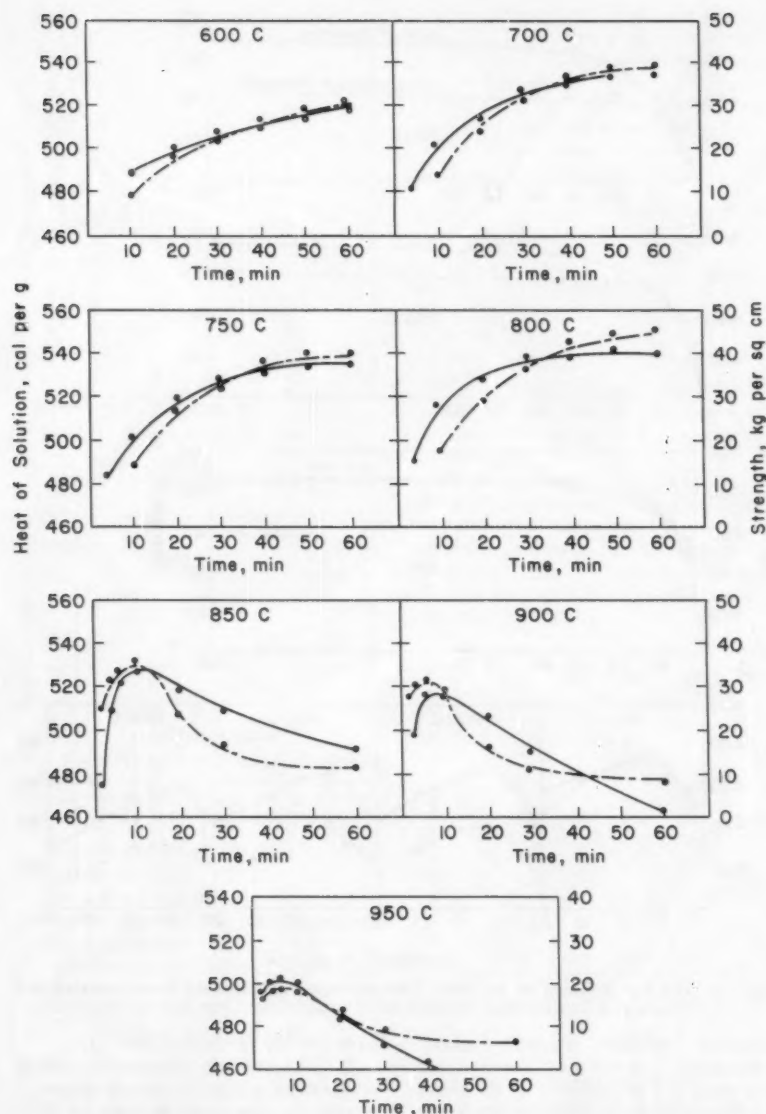


Fig. 13.—Heat of Solution of a Bentonite Activated at Different Temperatures and Times, and Compressive Strengths of Activated Bentonite-Lime Specimens.

strength of the activated clay-lime mixtures. This will provide the best activation conditions to obtain artificial pozzolans for the manufacture of pozzolanic cements or more economical pozzolan binders.

Although the activation curves as a function of time at constant temperature have not been determined for kaolinitic clays, from Figs. 4 and 6 it is concluded that the activation curves, as a function of temperature at constant time (1 hr), are similar in a general way to the other clay families and it is assumed that the activation process is of the same type. This would justify the projection of the above-mentioned to clays in general.

#### Conclusions

From this work the following conclusions are drawn:

1. The ability of different clay samples belonging to the same mineralogic family of being transformed into artificial pozzolans depends upon their lattice-water content.
2. As a routine test for determining the lattice water content of clays, determination with the Penfield tube on desiccated samples at 300 C, is proposed.
3. For a specific clay, the heat of solution and the strength of the activated clay-lime mixtures as a function of the activation temperature, present a

maximum for the same temperature.

4. For a specific clay, the heat of solution curves and strengths of the activated lime-clay mixtures as a function of activation time, at constant temperature (considered as parameter), show an asymptote parallel to the X axis up to a determined temperature. The maximum which never exceeds the value of the asymptote of the activation curve corresponds to that limit temperature. This temperature is the most ideal activation temperature.

5. A method is proposed to determine rapidly the optimum conditions for activating clay by determining the heat of solution curves of the thermally activated samples, as a function of the treatment time for different temperatures. Those conditions which determine a maximum heat of solution for the clay are the most advantageous for transforming them into artificial pozzolans, for use in the preparation of binding mixtures with lime, or for use in the manufacture of pozzolanic cements. In practice it will be sufficient to determine first the heat of solution curve as a function of the activation temperature at constant time (1 hr), and later fix the activation time at the temperature which produces the maximum of the previous curve according to the time-heat of solution curve (at constant temperature).

This survey was made at the "Laboratorio Central de Ensayo de Materiales de Construcción de la Escuela de Ingenieros de Caminos" in Madrid (Spain).

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# The Successive Determination of Manganese, Sodium, and Potassium in Cement by Flame Photometry

By C. L. FORD

The flame photometric methods of previous investigators have been simplified to permit the successive determination of manganese, sodium, and potassium in cement using only one sample and one set of standard solutions, the latter being the novel feature. The Beckman model DU spectrophotometer with flame and photomultiplier attachments and the Perkin-Elmer flame photometers, were used in the study. It was found that only the former could be used for manganese but that either could be used for sodium and potassium. The effect of interfering elements is discussed. The removal of silica from solutions of cement samples was found to be unnecessary under the test conditions. Appended is a "Method of Analysis of Cement for Manganic Oxide, Sodium Oxide, and Potassium Oxide by Flame Photometry," as developed.

**T**HE OBJECT of this investigation was to find if manganese, sodium, and potassium could be determined by flame photometry in one solution of a sample of cement with the use of only one set of standard solutions for the preparation of calibration curves. Diamond (1)<sup>1</sup> has reported a method in which the same sample solution was used for the determination of the above elements but with one set of standard solutions required for manganese and another for the alkali metals.

The problems involved in this study included the following:

1. Choice of instrument: Beckman model DU spectrophotometer No. 9200 flame and No. 4300 photomultiplier attachments, or Perkin-Elmer model 146 flame photometer.
2. A source of manganese for standardization.
3. The effect of the presence in the standard solutions of (a) alkalis on the manganese luminosity and (b) manganese on the alkali luminosity.
4. Accuracy of the flame photometer method as compared with the ASTM referee method for manganic oxide ( $Mn_2O_3$ ).
5. Comparison of alkali values by the Beckman DU spectrophotometer with those by the Perkin-Elmer (ASTM Method C 114-58 T)<sup>2</sup> flame photometer, and
6. The effect of removal of silica from the cement samples.

**NOTE.—DISCUSSION OF THIS PAPER IS INVITED,** either for publication or for the attention of the author. Address all communications to ASTM Headquarters, 1916 Race St., Philadelphia 3, Pa.

<sup>1</sup> The boldface numbers in parentheses refer to the list of references appended to this paper.

<sup>2</sup> Tentative Methods of Chemical Analysis of Portland Cement (C 114-58 T), 1958 Book of ASTM Standards, Part 4, Sections 15 to 18.

<sup>3</sup> Methods of Chemical Analysis of Portland Cement (C 114-53), 1958 Book of ASTM Standards, Part 4, Sections 26 to 27.

## Selection of Instruments

It was soon determined that the Perkin-Elmer instrument was not sufficiently sensitive, even with an acetylene flame, for the concentrations of manganese used in these tests; hence all determinations of manganese were made with the Beckman DU spectrophotometer. Alkalies were determined with both the Perkin-Elmer and the Beckman DU.

## Selection of a Source of Manganese

It was necessary for the preparation of the standard solutions to have a source of manganese of known purity. Samples of "pure" manganese metal from two different suppliers were found to contain only 94 to 96 per cent manganese. A sample of manganese sulfate ( $MnSO_4 \cdot H_2O$ ) obtained from J. T. Baker Chemical Co. was also tested. This material was said by the manufacturer to assay a minimum of 98 per cent. Analysis of both the oven-dried salt and the stock solution prepared therefrom showed that it contained 97.7 per cent of the theoretical content of manganese. Analyses were made by the ASTM referee method for manganic oxide in cement, the bismuthate method, ASTM Method C 114-53,<sup>3</sup> as were all subsequent referee analyses of cement samples of manganese. This manganese sulfate was used in this study in the preparation of all standard solutions.

## Manganese Luminosity

### Effect of Alkalies

All standard solutions (except those used for adjusting the wavelength dial) contained calcium equivalent to 6300 ppm of calcium oxide to compensate for the effect of calcium in the cement samples. Roy (2) found that potassium interfered with manganese when using the Beckman model B flame photom-

eter because the instrument did not resolve the potassium line at 404.5 m $\mu$  and the manganese line at 403.3 m $\mu$ . Diamond (1), however, found that the Beckman DU satisfactorily separated the lines. This was confirmed by the following tests which show the interference of potassium to the negligible. The effect of sodium as well as potassium on the luminosity of manganese using the Beckman DU was tested by preparing a series of solutions each containing manganese equivalent to 50 ppm manganic oxide, and calcium. These solutions also contained sodium and potassium equivalent to 0, 10, 25, 50, 75, and 100 ppm each of the respective oxides. The wavelength dial was set at the point of maximum luminosity at about 403.3 m $\mu$  with a solution containing manganese sulfate only. The transmittance dial was arbitrarily set at 50 with the 0 alkali solution. Transmittance dial readings were then made on the other solutions with the results shown in Table I. The average variation of the solutions containing alkalies was less than 0.1 dial unit from the setting of the 0 alkali solution. This corresponded to less than 0.1 ppm, or 0.001 per cent manganic oxide in a solution containing 1 g of cement per 100 ml. Hence, the interference or background effect of alkalies in the standard solutions had a negligible effect on the luminosity of manganese.



C. L. FORD, analytical consultant, Research and Development Division, Portland Cement Assn. has been employed by this Association for the past thirty-six years as, successively, chemist, chief of Analytical Laboratories, and consultant. His work has included studies of methods for chemical analysis and for the determination of the fineness and density of portland cement. Recent studies have been concerned with flame photometric methods for the analysis of cement.



TABLE I.—EFFECT OF SODIUM, POTASSIUM (AND CALCIUM) ON MANGANESE LUMINOSITY WITH BECKMAN MODEL DU FLAME PHOTOMETER.

Solution	Solution Content, ppm				Transmittance Dial Units
	Mn <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	CaO	
No. 1.....	50	0	0	6300	50.0 <sup>a</sup>
No. 2.....	50	100	100	6300	50.1
No. 3.....	50	75	75	6300	50.2
No. 4.....	50	50	50	6300	50.1
No. 5.....	50	25	25	6300	50.0
No. 6.....	50	10	10	6300	50.0

Average difference from solution No. 1 < +0.1

RELATIVE EFFECT OF CALCIUM BACKGROUND					
7	0	0	0	6300	5.0

<sup>a</sup> Transmittance dial arbitrarily set at 50 with solution without alkalis. Values shown are the average of two observations.

#### Effect of Calcium

The possible interference of calcium on manganese and sodium was largely negated by the use of 6300 ppm of calcium oxide in the standard solutions. This amount was equivalent to 63.0 per cent of calcium oxide in an average cement when the solution of the cement contained 1 g per 100 ml. The interference with manganic oxide due to the equivalent of 60.0 and 70.0 per cent calcium oxide (the approximate limits for portland cement composition) was found to cause variations from the 63.0 per cent calcium oxide solution of less than the equivalent of 0.01 per cent manganese oxide.

#### Effect of Magnesium

It has been reported by Dippel and Bricker (3) that magnesium interferes with manganese. Tests in these laboratories have confirmed that there is a little interference, but not to exceed an enhancement equivalent to about 0.01 per cent manganic oxide for average concentrations of manganic and manganese oxides. In one case of a solution containing relatively high concentrations of the oxides in amounts equivalent to 0.75 per cent manganic oxide and 5.00 per cent magnesium oxide, the manganic oxide value was raised by about 0.015 per cent over that of a similar solution containing no magnesium. This partially explains the somewhat high values shown in Table III for sample No. 19196—a sample found by gravimetric method to contain 0.93 per cent manganese oxide and (not shown in the table) 4.46 per cent magnesium oxide. For most cements, however, containing much lower percentages of manganese and magnesium, the effect of the latter is well within experimental error.

#### Background Effect at Manganese Wavelength

The background effect of the calcium at the above dial settings (see "Effect of Alkalies") was found with a solution

containing 6300 ppm calcium oxide only. As shown in Table I, the reading obtained was 5 dial units. In later work, with the transmittance set at 100 with solutions containing 100 ppm each of manganic, sodium, and potassium oxides, and 6300 ppm calcium oxide, numerous tests of the background effect of solutions containing only calcium oxide gave a reading of 6 dial units. This latter value was used as a zero correction for all manganic oxide values obtained in this investigation.

Diamond (1) used as a correction the background of cement at a wavelength of 401.0 mμ. Tests of five cements of varying compositions at this wavelength also showed readings of 6 dial units. This was the same as that obtained at a wavelength of 403.3 mμ with the solution containing 6300 ppm calcium oxide only. Thus by using the latter solution for the background or zero correction the need for

shifting the wavelength dial was eliminated. Apparently the background interference at 403.3 mμ is due to calcium rather than any other element in cement.

#### Effect of Manganese on Alkali Luminosity

The above set of standard solutions was also used to find the effect of manganese on the sodium and potassium oxide dial readings with the Beckman DU. The values obtained by averaging two or more readings are shown in Table II. It will be observed that the average differences between the solutions of a given concentration, with and without manganese, were all less than one dial unit. The grand average difference for sodium oxide was +0.2 and for potassium oxide was less than +0.1 dial unit. These calculated values are within the limit of accuracy of the instrument.

Tests for the effect of manganese using the Perkin-Elmer flame photometer were made with a set of manganese-alkali standards solutions prepared for the actual analysis of cement samples. They differed from those used above only in that the manganic oxide content varied as shown in Table II, along with the alkali content, from 0 to 100 ppm instead of being constant at 50 ppm manganic oxide. The results, also shown in Table II, are believed to be comparable between instruments in spite of the difference in manganese content. The average variations between standards with and without manganese were slightly greater than with the Beckman DU, but still

TABLE II.—EFFECT OF MANGANESE (AND CALCIUM) ON SODIUM AND POTASSIUM LUMINOSITY.

Solution Contents, ppm							Transmittance Dial Units <sup>a</sup>						
A			B				Na <sub>2</sub> O			K <sub>2</sub> O			
Na <sub>2</sub> O	K <sub>2</sub> O	CaO	Mn <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	CaO	A	B	Difference <sup>b</sup>	A	B	Difference	
BECKMAN MODEL DU FLAME PHOTOMETER													
100	100	6300	50	100	100	6300	100.0	100.5*	+0.5	100.0	100.2*	+0.2	
75	75	6300	50	75	75	6300	87.0	87.2	+0.2	80.0	80.0	0	
50	50	6300	50	50	50	6300	69.0	69.7	+0.7	58.0	58.1*	+0.1	
25	25	6300	50	25	25	6300	46.0	46.0	0	33.0	33.0*	0	
10	10	6300	50	10	10	6300	22.0	22.1	+0.1	15.0	15.0*	0	
0	0	6300	50	0	0	6300	3.0	2.9	-0.1	0	0	0	
Grand average difference									+0.2	< +0.1			
PERKIN-ELMER MODEL 146 FLAME PHOTOMETER													
100	100	6300	100	100	100	6300	100.0	100.0	0	100.0	100.7	+0.7	
75	75	6300	75	75	75	6300	84.0	84.5	+0.5	79.0	79.7	+0.7	
50	50	6300	50	50	50	6300	62.5	62.5*	0	55.5	56.0	+0.5	
25	25	6300	25	25	25	6300	32.5	33.0	+0.5	28.5	29.2	+0.7	
10	10	6300	10	10	10	6300	14.0	14.5	+0.5	11.0	11.0	0	
0	0	6300	0	0	0	6300	0	0	0	0	0	0	
Grand average difference									+0.3	+0.5			

<sup>a</sup> All values are the average of two or three observations. (Three observations were made for all values, but in some instances some individual values were obviously in error because of instrument fluctuations; hence the values marked with an asterisk\* are the average of only two observations. The grand average differences are weighted values.)

<sup>b</sup> Differences between column B and column A readings.

less than one dial unit. The grand average difference for sodium oxide was +0.3 and for potassium oxide was +0.5 unit. These, again, are within the limits of accuracy of the instrument. It was concluded that the presence of manganese had an insignificant effect on the luminosity of sodium and potassium when either instrument was used.

## Experimental Program

### Preparation

Ten cements were selected for the flame photometric determination of manganese, sodium, and potassium. They were selected to give a considerable range of composition, not only of the above elements but of the other constituents of cement. Included were National Bureau of Standards sample No. 177, four Portland Cement Assn. check samples, one portland-slag cement and four miscellaneous type I cements. The set of standard solutions, referred

to in the preceding paragraph, was used for the preparation of calibration curves. In addition, a solution containing 100 ppm each of manganese, sodium, and potassium (calculated as the oxides), but no calcium, was prepared for setting the wavelength selector to maximum response. A more detailed description of the preparation of the solutions, and other steps in the method as developed, are presented in the Appendix.

The cements (except No. 177) were analyzed for manganese oxide by the ASTM referee Method C 114-53.<sup>3</sup> They had been analyzed previously for sodium and potassium oxides by the ASTM flame photometer Method C 114-58 T<sup>2</sup> using a Perkin-Elmer model No. 52A instrument. The values obtained are shown in Table III. The values for the NBS No. 117 sample are those certified by the National Bureau of Standards.

### Manganese

As stated above, the Perkin-Elmer flame photometer lacked sufficient sensitivity for the determination of manganese in cement; hence, the Beckman DU spectrophotometer with flame and photomultiplier attachments was used in all the tests. The latter was operated according to the manufacturer's instructions (4). An oxyhydrogen flame was used. The wavelength dial was adjusted with the solution containing no calcium to the point of maximum luminosity at about 403.3 mμ. The instrument was set for the blue tube, slit width 0.01 mm, selector at 0.1, and photomultiplier on "full." (It was found that the emission of manganese was too low to be determined, even with a full slit width of 2.0 mm, without the use of the photomultiplier.)

A standard calibration curve was prepared from transmittance dial readings obtained by setting the dial at

TABLE III.—COMPARISON OF PROPOSED FLAME PHOTOMETER METHODS WITH ASTM METHODS.

Cement		Method											
Number	Type	ASTM		Beckman DU with Mn <sub>2</sub> O <sub>3</sub> , Alkali Standards									
		Ref- eree <sup>a</sup>	Flame Photom- eters <sup>a,b</sup>	With SiO <sub>2</sub>				Without SiO <sub>2</sub>					
				Test			Aver- age	Differ- ence <sup>d</sup>	Test		Aver- age	Differ- ence	
				1	2	3			1	2			
MANGANIC OXIDE (Mn <sub>2</sub> O <sub>3</sub> ), PER CENT													
B. of S. 177...	...	0.05 <sup>c</sup>	...	0.05	0.05	0.05	0.05	0	0.07	0.07	0.07	+0.02	
PCA Ck 1...	I	0.29	...	0.31	0.30	0.30	0.30	+0.01	0.32	0.33	0.33	+0.04	
PCA Ck 2...	I	0.04	...	0.04	0.04	...	0.04	0	0.05	0.05	0.05	+0.01	
PCA Ck 3...	I	0.09	...	0.10	0.10	...	0.10	+0.01	0.11	0.12	0.12	+0.03	
PCA Ck 4...	II	0.05	...	0.05	0.05	...	0.05	0	0.07	0.07	0.07	+0.02	
LTS 18...	I	0.45	...	0.49	0.48	...	0.49	+0.04	0.48	0.48	0.48	+0.03	
19196...	IS <sup>e</sup>	0.93	...	0.98	0.98	...	0.98	+0.05	0.97	0.97	0.97	+0.04	
19269...	I	0.58	...	0.61	0.63	0.62	0.62	+0.04	0.60	0.60	0.60	+0.02	
19270...	I	0.25	...	0.27	0.28	0.26	0.27	+0.02	0.27	0.27	0.27	+0.02	
19272...	I	0.18	...	0.19	0.20	0.19	0.19	+0.01	0.20	0.20	0.20	+0.02	
Average								+0.018				+0.025	
SODIUM OXIDE (Na <sub>2</sub> O), PER CENT													
B. of S. 177...	...	0.014 <sup>c</sup>	...	0.14	0.14	0.14	0.14	0	0.15	0.14	0.15	+0.01	
PCA Ck 1...	I	0.25	...	0.26	0.27	...	0.27	+0.02	0.26	0.26	0.26	+0.01	
PCA Ck 2...	I	0.06	...	0.07	0.06	...	0.07	+0.01	0.07	0.07	0.07	+0.01	
PCA Ck 3...	I	0.26	...	0.29	0.29	...	0.29	+0.03	0.30	0.29	0.30	+0.04	
PCA Ck 4...	II	0.60	...	0.62	0.62	...	0.62	+0.02	0.63	0.62	0.63	+0.03	
LTS 18...	I	0.15	...	0.15	0.16	...	0.16	+0.01	0.15	0.15	0.15	0	
19196...	IS <sup>e</sup>	0.12	...	0.14	0.16	...	0.15	+0.03	0.15	0.15	0.15	+0.03	
19269...	I	0.19	...	0.12	0.10	...	0.11	+0.02	0.12	0.12	0.12	+0.03	
19270...	I	0.34	...	0.36	0.36	...	0.36	+0.02	0.36	0.35	0.36	+0.02	
19272...	I	0.31	...	0.32	0.32	...	0.32	+0.01	0.32	0.32	0.32	+0.01	
Average								+0.017				+0.019	
POTASSIUM OXIDE (K <sub>2</sub> O), PER CENT													
B. of S. 177...	...	0.56 <sup>c</sup>	...	0.56	0.55	0.56	0.56	0	0.55	0.54	0.55	-0.01	
PCA Ck 1...	I	0.59	...	0.59	0.59	...	0.59	0	0.58	0.58	0.58	-0.01	
PCA Ck 2...	I	0.25	...	0.25	0.25	...	0.25	0	0.24	0.24	0.24	-0.01	
PCA Ck 3...	I	0.70	...	0.67	0.71	...	0.69	-0.01	0.67	0.68	0.68	-0.02	
PCA Ck 4...	II	0.14	...	0.14	0.14	...	0.14	0	0.14	0.14	0.14	0	
LTS 18...	I	0.14	...	0.14	0.14	...	0.14	0	0.15	0.14	0.15	+0.01	
19196...	IS <sup>e</sup>	0.35	...	0.35	0.34	...	0.35	0	0.34	0.34	0.34	-0.01	
19269...	I	0.15	...	0.14	0.14	...	0.14	-0.01	0.14	0.14	0.14	-0.01	
19270...	I	0.58	...	0.56	0.57	...	0.57	-0.01	0.57	0.56	0.57	-0.01	
19272...	I	0.61	...	0.58	0.61	...	0.60	-0.01	0.59	0.59	0.59	-0.02	
Average								-0.004				-0.009	

<sup>a</sup> B. of S. No. 177 values are those certified by the Bureau of Standards; PCA check samples values for Na<sub>2</sub>O and K<sub>2</sub>O are the average of duplicate analyses by two or more analysts; all others are the average of two or more analyses by one analyst.

<sup>b</sup> By Perkin-Elmer model 52A.

<sup>c</sup> Certified by Bureau of Standards.

<sup>d</sup> Differences from values obtained by ASTM methods.<sup>3,5,6</sup>

<sup>e</sup> IS = Portland slag.

100 with the manganese-alkali solution containing 100 ppm each of manganic, sodium, and potassium oxides, then determining the dial readings of the other concentrations. The zero reading of 6 dial units, as previously stated, was used as a correction before plotting the other dial readings, and as a correction for the cement sample readings. A calibration curve for manganic oxide is illustrated in Fig. 1.

In the first part of this work, hydrochloric acid solutions of the cements were used after removal of the small acid-insoluble portion by filtration. In later tests, silica was removed before analysis by a single evaporation of hydrochloric acid solutions of the cements followed by redissolving and filtration. Silica was removed to investigate its effect, if any, on the

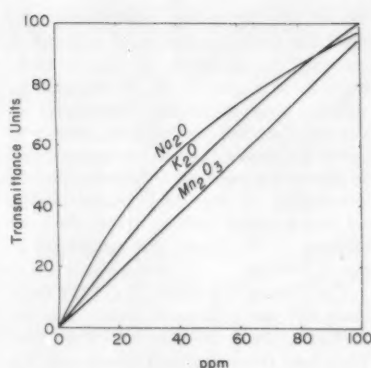


Fig. 1.—Calibration curves of the transmittance of manganese, sodium, and potassium emission with the Beckman model DU flame apparatus.

emission of manganese since silica, the largest constituent of cement next to calcium oxide, is not compensated for in the standard solutions. The possible interference of minor constituents was not studied.

The individual percentage values, the average values, and the differences from the values by the ASTM method,<sup>2,3</sup> both "with SiO<sub>2</sub>" and "without SiO<sub>2</sub>" are shown in Table III. It will be observed that either "with" or "without SiO<sub>2</sub>" in the solutions, the flame photometer values for manganic oxide tended to be slightly higher than the volumetric (ASTM referee method<sup>2,3</sup>) values, but the average difference "with SiO<sub>2</sub>" was only +0.018 per cent. This is considered to be a favorable relationship. "Without SiO<sub>2</sub>" the average difference was 0.023 per cent,

TABLE IV.—COMPARISON OF FLAME PHOTOMETER ALKALI VALUES OBTAINED BY THE USE OF Mn<sub>2</sub>O<sub>3</sub>-ALKALI STANDARDS WITH THOSE OBTAINED BY THE ASTM METHOD.

Cement		Flame Photometer														
		Using Mn <sub>2</sub> O <sub>3</sub> -Alkali Standards														
		ASTM Method C 114 <sup>a</sup>	Beckman DU				Perkin-Elmer 146									
			With SiO <sub>2</sub>		Without SiO <sub>2</sub>		With SiO <sub>2</sub>				Without SiO <sub>2</sub>					
Number	Type		Average <sup>b</sup>	Difference <sup>c</sup>	Average <sup>b</sup>	Difference <sup>c</sup>	Test			Average	Difference	Test		Average	Difference	
		1					2	3	1			2				
SODIUM OXIDE (Na <sub>2</sub> O), PER CENT																
B. of S. 177...	...	0.14	0.14	0	0.14	0	0.12	0.11	0.12	0.12	-0.02	0.13	0.13	0.13	-0.01	
PCA Ck 1...	I	0.25	0.27	+0.02	0.26	+0.01	0.24	0.23	0.23	0.23	-0.02	0.25	0.25	0.25	0	
PCA Ck 2...	I	0.06	0.07	+0.01	0.07	+0.01	0.05	0.05	...	0.05	-0.01	0.07	0.07	0.07	+0.01	
PCA Ck 3...	I	0.26	0.29	+0.03	0.29	+0.03	0.25	0.25	...	0.25	-0.01	0.27	0.27	0.27	+0.01	
PCA Ck 4...	II	0.60	0.62	+0.02	0.63	+0.03	0.59	0.59	...	0.59	-0.01	0.62	0.60	0.61	+0.01	
LTS 18...	I	0.15	0.16	+0.01	0.15	0	0.13	0.13	...	0.13	-0.02	0.15	0.14	0.15	0	
19196...	IS <sup>d</sup>	0.12	0.15	+0.03	0.15	+0.03	0.11	0.11	...	0.11	-0.01	0.13	0.13	0.13	+0.01	
19269...	I	0.09	0.11	+0.02	0.12	+0.03	0.09	0.09	0.10	0.09	0	0.11	0.11	0.11	+0.02	
19270...	I	0.34	0.36	+0.02	0.36	+0.02	0.33	0.32	0.33	0.33	-0.01	0.35	0.35	0.35	+0.01	
19272...	I	0.31	0.32	+0.01	0.32	+0.01	0.29	0.30	0.31	0.30	-0.01	0.31	0.32	0.32	+0.01	
Average				+0.017		+0.017						0.012				+0.007
POTASSIUM OXIDE (K <sub>2</sub> O), PER CENT																
B. of S. 177...	...	0.56	0.56	0	0.55	-0.01	0.57	0.57	0.57	0.57	+0.01	0.52	0.53	0.53	-0.03	
PCA Ck 1...	I	0.59	0.59	0	0.58	-0.01	0.60	0.60	0.61	0.60	+0.01	0.56	0.56	0.56	-0.03	
PCA Ck 2...	I	0.25	0.25	0	0.24	-0.01	0.25	0.25	...	0.25	0	0.24	0.23	0.24	-0.01	
PCA Ck 3...	I	0.70	0.69	-0.01	0.68	-0.02	0.71	0.70	...	0.71	+0.01	0.66	0.66	0.66	-0.04	
PCA Ck 4...	II	0.14	0.14	0	0.14	0	0.14	0.14	...	0.14	0	0.13	0.13	0.13	-0.01	
LTS 18...	I	0.14	0.14	0	0.15	+0.01	0.14	0.14	...	0.14	0	0.13	0.13	0.13	-0.01	
19196...	IS <sup>d</sup>	0.35	0.35	0	0.34	-0.01	0.36	0.36	...	0.36	+0.01	0.34	0.34	0.34	-0.01	
19269...	I	0.15	0.14	-0.01	0.14	-0.01	0.14	0.14	0.14	0.14	-0.01	0.13	0.13	0.13	-0.02	
19270...	I	0.58	0.57	-0.01	0.57	-0.01	0.59	0.59	0.58	0.59	+0.01	0.56	0.56	0.56	-0.02	
19272...	I	0.61	0.60	-0.01	0.59	-0.02	0.61	0.60	0.60	0.60	-0.01	0.56	0.56	0.56	-0.05	
Average				-0.004		-0.009						+0.003				-0.023
SODIUM OXIDE (Na <sub>2</sub> O) PLUS POTASSIUM OXIDE (K <sub>2</sub> O) AS SODIUM OXIDE (Na <sub>2</sub> O) <sup>e</sup> , PER CENT																
B. of S. 177...	...	0.51	0.51	0	0.50	-0.01	...	...	...	0.50	-0.01	...	...	0.48	-0.03	
PCA Ck 1...	I	0.64	0.66	+0.02	0.64	0	...	...	...	0.62	-0.02	...	...	0.62	-0.02	
PCA Ck 2...	I	0.22	0.23	+0.01	0.23	-0.01	...	...	...	0.21	-0.01	...	...	0.23	+0.01	
PCA Ck 3...	I	0.72	0.74	+0.02	0.74	+0.02	...	...	...	0.72	0	...	...	0.70	-0.02	
PCA Ck 4...	II	0.69	0.71	+0.02	0.72	+0.03	...	...	...	0.68	-0.01	...	...	0.70	+0.01	
LTS 18...	I	0.24	0.25	+0.01	0.25	+0.01	...	...	...	0.22	-0.02	...	...	0.24	0	
19196...	IS <sup>d</sup>	0.35	0.38	+0.03	0.37	+0.02	...	...	...	0.35	0	...	...	0.35	0	
19269...	I	0.19	0.20	+0.01	0.21	+0.02	...	...	...	0.18	-0.01	...	...	0.20	+0.01	
19270...	I	0.62	0.74	+0.02	0.74	+0.02	...	...	...	0.72	0	...	...	0.72	0	
19272...	I	0.71	0.71	0	0.71	0	...	...	...	0.69	-0.02	...	...	0.69	-0.02	
Average				+0.014		+0.012						-0.010				-0.006

<sup>a</sup> B. of S. No. 177 values are those certified by the Bureau of Standards; PCA check samples are the average of duplicate analyses by two or more analysts and all others are the average of two or more analyses by one analyst, using Perkin-Elmer model 52A.

<sup>b</sup> For individual values see Table III.

<sup>c</sup> Differences from values by ASTM methods.<sup>2,3,4</sup>

<sup>d</sup> IS = Portland slag.

<sup>e</sup> Na<sub>2</sub>O + 0.658 × K<sub>2</sub>O.



very slightly higher. The results of the above tests showed that there is nothing to be gained by taking the additional time required to remove silica. The manganic oxide results were very satisfactory with samples prepared "with SiO<sub>2</sub>" by the current ASTM method<sup>2</sup> for the flame photometric analysis for alkalis.

#### Alkalis by Beckman DU

After determining manganic oxide, the same samples and standards were used to determine alkalis with the Beckman DU. The instrument settings were as follows:

	Wavelength, mμ	Tube	Slit width, mm	Selector Switch	Photomultiplier Switch
Sodium oxide.....	589	Blue	0.01	0.1	2
Potassium oxide....	768	Red	0.05	0.1	Off

The object of the tests was to determine the following as compared to previously determined values by the ASTM method: (1) the effect of the manganese-alkali solutions, (2) the effect of the Beckman DU instead of Perkin-Elmer, and (3) the effect of silica removal.

The results of the individual analyses, the averages, and the differences from the ASTM method values are shown in Table III. Alkali calibration curves obtained with the Beckman DU are shown in Fig. 1.

As shown, the sodium oxide values were slightly higher with an average difference of +0.017 per cent both "with" and "without SiO<sub>2</sub>." These higher values are believed to be due to the instrument rather than to the use of the manganese-alkali standards because, as will be shown later, the results were not higher when the Perkin-Elmer flame photometer was used. According to Diamond and Bean (5) removal of silica results in higher sodium oxide values. However, the author used a smaller effect band width (about 0.3 mμ using a slit width of 0.01 mm) than any they used. Most of their differences between samples prepared "with" and "without SiO<sub>2</sub>" occurred with effective band widths varying from 1 to 30 mμ, the significant differences occurring with the larger effective band widths. The results for sodium oxide shown in Table III indicated that when a small effective band width was used, there was no advantage in removing silica.

As regards the potassium oxide values, it will be observed from Table III that the difference between results by the methods described above and by the ASTM methods, both "with" and "without SiO<sub>2</sub>," averaged less than

0.01 per cent which is within the limits of accuracy of the instrument. Removal of silica showed very slightly lower results with average differences of -0.009 versus -0.004 per cent.

#### Alkalis by Perkin-Elmer

Having found that the manganese-alkali standards could be used satisfactorily with the Beckman DU for the determination of manganic, sodium, and potassium oxides, the use of these same standards for the Perkin-Elmer model 146 was investigated. The same (or similarly prepared) cement sample solutions were used for both instruments.

The latter instrument was operated according to the ASTM Method C 114-58 T.<sup>2</sup>

The individual and the average values obtained with the Perkin-Elmer model 146, together with the differences from the values obtained with the Perkin-Elmer model 52A by the ASTM method, are presented in Table IV. For comparison, the average values and the differences observed with the Beckman DU are repeated from Table III.

It may be seen from Table IV that the sodium oxide values differed by only 0 to 0.02 per cent from the ASTM method values with average differences of about 0.01 per cent. It is not known whether the slightly lower average difference of -0.012 per cent "with SiO<sub>2</sub>" was due to the difference in Perkin-Elmer models or to the use of the manganese-alkali standards. In either case it was negligible. The higher average difference "without SiO<sub>2</sub>" (+0.007 as compared to -0.012 per cent) is believed to be due to the effect of a larger effective band width as discussed by Diamond and Bean (5). The Perkin-Elmer instruments have a fixed slit width (which determine the effective band width), and no attempt was made to measure or alter it. As suggested previously, the very narrow effective band width used with the Beckman DU apparently accounted for the somewhat higher average differences (+0.017) and the lack of any difference between values obtained with and without removal of silica before test.

The potassium oxide "with SiO<sub>2</sub>" values differed by only 0 to 0.01 per cent from the ASTM method values, with a negligible average difference of +0.003 per cent. The "without SiO<sub>2</sub>" values were appreciably lower with

correspondingly greater differences of -0.01 to -0.05 per cent and an average difference of -0.023 per cent. The cause of these lower values was not investigated. The significant fact so far as this study is concerned is that potassium oxide determined "with SiO<sub>2</sub>" according to the ASTM procedure, except for the use of the manganese-alkali standards, gave satisfactory results with either instrument.

The total alkali contents of the cements, calculated as sodium oxide (Na<sub>2</sub>O+0.658 × K<sub>2</sub>O), are shown at the bottom of Table IV. This tabulation shows that the average difference between the various experimental methods and the ASTM method values was only about +0.01 per cent. In other words, for total alkalis the average effect of using manganese-alkali standards with either the Beckman DU or the Perkin-Elmer model 146, with or without removal of silica, did not cause an average variation from the ASTM values much in excess of 0.01 per cent. The differences for any of the cements tested did not exceed 0.03 per cent.

It has no doubt been noted that all the comparisons of alkali values have been made with those obtained with the ASTM flame photometric method<sup>2</sup> rather than the referee gravimetric method.<sup>4</sup> This was so because the author was primarily concerned with flame photometric methods. As previously stated, the object of the study was to develop a simple method for manganese using standard solutions which could be used also in the determination of sodium and potassium. The study was not concerned with the relative accuracy of the gravimetric and photometric methods for alkalis, but only with the relationship between different photometric methods. Therefore, since the current ASTM photometric procedure applies specifically to the Perkin-Elmer instrument, alkali results obtained therewith, using the prescribed procedure, were used as the basis of comparison of the variations in the procedure described above.

#### Summary

It has been shown that manganese in cement can be determined flame-photometrically using the Beckman model DU spectrophotometer with No. 9200 flame and No. 4300 photomultiplier attachments and preparing calibration curves with solutions containing known amounts of manganese plus calcium equivalent to 6300 ppm of calcium oxide. By inclusion of known amounts of sodium and potassium in the above standard solutions, the same solutions may be used also to obtain data for calibration curves for the determination

<sup>4</sup> *Ibid.*, Sections 21-22.

of these latter elements. The analyses for sodium and potassium may be made on the cement solutions prepared for the determination of manganese using either the Beckman DU or the Perkin-Elmer model 146 flame photometer. It was found unnecessary to separate anything from the solutions of cement samples except the small amount of hydrochloric acid-insoluble material that had to be removed by filtration to obtain clear solutions.

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- (4) Beckman Instruments, Inc., *Bulletin Nos. 305-A and 334-A*, "Instruction Manual."
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## APPENDIX

### METHOD OF ANALYSIS OF CEMENT FOR MANGANIC OXIDE, SODIUM OXIDE, AND POTASSIUM OXIDE BY FLAME PHOTOMETRY

#### Scope

1. This method of analysis is intended for successively determining the manganic, sodium, and potassium oxide contents of cement by flame photometry using one solution of a sample and one set of standard solutions.

#### Apparatus

2. (a) *For Manganic Oxide*.—A Beckman model DU spectrophotometer with model 9200 flame attachment, oxyhydrogen burner, and photomultiplier.

(b) *For Sodium Oxide and Potassium Oxide*.—Either the apparatus described in Paragraph (a) or a Perkin-Elmer model 52 or 146 flame photometer with an air-propane burner.

(c) Cylinders of compressed oxygen, hydrogen, and propane and a source of compressed air, with reducing valves.

#### Reagents

3. (a) *Calcium Chloride Stock Solution*.—Add 300 ml of water to 112.5 g of calcium carbonate ( $\text{CaCO}_3$ ) (Note 1) in a 1500 or 2000-ml beaker. While stirring, slowly add 500 ml of hydrochloric acid (specific gravity 1.18). Cool the solution to room temperature filter into a 1-liter volumetric flask, dilute to the mark, and mix thoroughly. This solution contains the equivalent of 63,000 ppm (6.3 per cent) of  $\text{CaO}$ .

NOTE 1.—The calcium carbonate shall be reagent grade low-alkali  $\text{CaCO}_3$  carrying a specification limit of 0.020 per cent total alkalies as sulfate. The purchaser should assure himself that this specification is met.

(b) *Manganese-Alkali Stock Solution*.—Dissolve in water 1.8858 g of reagent grade sodium chloride ( $\text{NaCl}$ ), 1.5830 g of reagent grade potassium chloride ( $\text{KCl}$ ), and 2.1412 g (or the proportionate amount as determined by analysis, see Note 2) of reagent grade manganese sulfate ( $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ ) (all previously dried at 105°C for several hours, Note 3), dilute to 1 liter in a volumetric flask, and mix thoroughly. This solution contains the equivalent of 1000 ppm (0.10 per cent) each of  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ , and  $\text{Mn}_2\text{O}_3$ .

NOTE 2.—The  $\text{MnSO}_4 \cdot \text{H}_2\text{O}$  may not be sufficiently pure to use the theoretical weight of 2.1412 g (equivalent to 1.0000 g of  $\text{Mn}_2\text{O}_3$ ). Before use in the stock solution, prepare a

trial solution containing 2.1412 g per liter. Analyze 5 and 10-ml aliquots of this solution for  $\text{Mn}_2\text{O}_3$  according to ASTM Method C 114 - 53, sections 26 and 27.<sup>3</sup> Correct the weight of  $\text{MnSO}_4 \cdot \text{H}_2\text{O}$  used in the stock solution so that the latter will contain the equivalent of 1.0000 g  $\text{Mn}_2\text{O}_3$ .

NOTE 3.—The temperature must be kept well below 117°C, the temperature at which  $\text{MnSO}_4 \cdot \text{H}_2\text{O}$  becomes unstable.

(c) *Standard Solutions*.—Prepare the standard solutions prescribed in Table V. The required volume of manganese-alkali stock solutions shall be measured in calibrated pipets or burets and the  $\text{CaCl}_2$  stock solution may be measured in a graduated cylinder. Each solution shall be placed in a volumetric flask, diluted to the volume indicated in Table V and mixed thoroughly. The solutions shall be stored in bottles of acid-resistant glass with ground-glass or rubber stoppers.

#### Calibration of Apparatus

4. (a) *General*.—The instructions given below apply to the Beckman model DU spectrophotometer with accessories. For the preparation and calibration of the Perkin-Elmer flame photometer, follow the method prescribed in ASTM Method

C 114 - 58 T, section 17.<sup>2</sup> For a description of and further details of operation of the Beckman DU other than described below, refer to Beckman Instruments, Inc., *Bulletins 305-A and 334-A*. Further, some of the instructions which follow for settings and adjustments may have to be varied with different instruments. The manufacturer's instructions for oxygen and hydrogen pressures for the burner used should be followed. Slit width and photomultiplier settings may have to be varied with different burners.

(b) *Warmup of Apparatus and Adjustment of Flame*.—Set the selector switch to 0.1 and select the proper phototube, resistor, and photomultiplier setting (see Table VI). Allow the electrical system about 1 hr to warm up. See that the shutter switch is off and that the fuel and oxygen regulators on the control panel are closed. At the end of the warmup period, open the tank valves and adjust the pressure regulators to about 30 psi for oxygen and 10 psi for hydrogen. Next, open the oxygen regulator on the control panel to about 10 psi, then, while holding a match near to and just below the tip

TABLE V.—STANDARD SOLUTIONS.

Designation of Standard		Composition of Standard		Final Volume of Solution, ml
Solution	Concentration of Manganese and Alkali (Expressed as $\text{Mn}_2\text{O}_3$ , $\text{Na}_2\text{O}$ , or $\text{K}_2\text{O}$ ), ppm	Calcium Chloride Stock Solution, ml	Manganese-Alkali Stock Solution, ml	
No. 1 . . . .	100	200	200	2000
No. 2 . . . .	75	100	75	1000
No. 3 . . . .	50	100	50	1000
No. 4 . . . .	25	100	25	1000
No. 5 . . . .	10	100	10	1000
No. 6 . . . .	0	100	0	1000
No. 7 . . . .	100	0 <sup>a</sup>	100	1000

<sup>a</sup> The calcium-free solution is used only in determining the correct positions of the wavelength selector for maximum responses to  $\text{Mn}_2\text{O}_3$ ,  $\text{Na}_2\text{O}$ , and  $\text{K}_2\text{O}$ .

TABLE VI.—INSTRUMENT SETTINGS FOR MANGANESE, SODIUM, AND POTASSIUM.

Metal	Wavelength, $\text{m}\mu$	Phototube	Load Resistor, megohms	Slit, mm	Selector Switch	Photomultiplier
Manganese . . . .	403.3	Blue	22	0.01	0.1	Full
Sodium . . . . .	589	Blue	22	0.01	0.1	2
Potassium . . . .	768	Red	10 000 <sup>a</sup>	0.05	0.1	Off

<sup>a</sup> With some instruments a 2000-megohm resistor may give better performance.

of the burner, open the hydrogen regulator to about 4 psi. Fill one of the 5-ml sample beakers with a solution containing sodium and move it under the capillary by rotating the sample positioner control. Adjust the visible yellow flame to a height of about 2½ in. by adjusting the fuel needle valve on the side of the pressure control panel and, if necessary, varying slightly the pressures shown above. Remove the beaker, replace with a beaker of distilled water, and allow the latter to atomize for 15 to 30 sec to prevent the formation of salt incrustations on the burner tip. *Important:* This must be done each time after any solution is used in the flame.

(c) *Calibration Procedure.*—

(1) Rotate the dark current control to zero the meter needle. Set the instrument for the element to be determined as shown in Table VI.

(2) Set the wavelength dial to the desired point for the element to be determined then check for maximum response as follows: set the transmittance dial at 100 per cent. Atomize the calcium-free standard solution (No. 7) into the flame, turn the shutter switch on, and adjust the meter needle to zero with the sensitivity control. Move the wavelength dial control slightly to either side until, after adjustment of the meter needle to zero (Note 4) further changing of the wavelength produces no movement of the needle to the left. Turn the shutter switch off, remove the solution and atomize distilled water. The above procedure must be followed for each element.

**NOTE 4.**—If it is impossible to zero the meter needle with the sensitivity control, it may be necessary to vary the slit widths from the values shown in Table VI.

(3) With the shutter closed, check the meter and, if necessary, zero the needle with the dark current control (Note 5). With the transmittance dial still set at 100, atomize into the flame the 100-ppm manganese-alkali-calcium standard solution (No. 1). Open the shutter and zero the meter needle with the sensitivity control.

**NOTE 5.**—If the needle has a tendency to drift with the dark current only, it should be zeroed (with the shutter closed) before each transmittance reading of a standard or sample solution.

(4) Next (after atomizing distilled water) atomize the 75-ppm standard solution (No. 2) and zero the needle with the transmittance dial. Check the reading of the 100-ppm solution and if it has changed from 100, reset the meter to zero with the sensitivity control. Repeat alternately

with the 100 and the 75-ppm solutions until there is less than one transmittance dial division change in the respective readings. Record the transmittance values as "gross luminosity."

(5) Repeat the above procedure with each of the other standard solutions containing calcium (Nos. 3, 4, 5, and 6) and record the readings as "gross luminosity" (Note 6).

**NOTE 6.**—The reading obtained for the solution containing calcium, but no manganese or alkalis (No. 6), represents the blank or background emission correction to be subtracted from all readings. This applies to solutions of cement as well as standard solutions since the enhancement of the emission of the above elements has been found to be due almost entirely to calcium.

(6) Correct the gross luminosity readings for the background emission. From the net luminosity values thus obtained, plot calibration curves for each oxide using cross-section paper of such type that each division on the ordinate represents a transmittance reading of one, and each division on the abscissa represents a concentration of 1 ppm or 0.01 per cent of manganic, sodium, or potassium oxide.

(d) *Shutdown of Apparatus.*—Shut down the instrument in the following order: close the fuel tank valve and regulator, the oxygen tank valve and regulator, then the regulators on the control panel. Since a leaky fuel valve may cause a small flame to burn after the valves are closed, always blow out the flame. Turn the selector and photomultiplier switches to "off."

**Procedure**

5. (a) *Solution of the Cement.*—Grind a representative sample of the cement to pass a 100-mesh sieve and remix thoroughly. Place 1.000 g of the sample into a 250-ml beaker and disperse with 20 ml of water, using a swirling motion of the beaker. While still swirling, add 5 ml of hydrochloric acid (sp gr 1.18) all at once. Break up with a rubber policeman any lumps of cement remaining undissolved. Dilute immediately with 25 ml of water, using the water to rinse the policeman. Bring to just short of boiling on a hot plate. Digest on a steam bath or low-temperature hot plate for 15 min and filter through a medium-texture paper into a calibrated 100-ml volumetric flask. Wash the beaker and paper thoroughly with water. Cool the contents of the flask to a temperature which shall closely approximate that of the standard solutions when the apparatus was calibrated, dilute to 100 ml, and mix the solution thoroughly.

(b) *Determination of  $Mn_2O_3$ .*—Warm up the apparatus, adjust the controls, light the flame, and find the point of maximum response to  $Mn_2O_3$  as described in Section 4, (a), (b) (1), and (b) (2). Atomize the 100-ppm manganese-alkali-calcium standard solution (No. 1) and, with the transmittance dial set at 100, zero the meter needle with the sensitivity control.

Following these adjustments, atomize the cement solution and note the transmittance reading after the meter needle has been brought to zero with the transmittance dial (Note 7). Select the standard solution closest to the cement solution in  $Mn_2O_3$  content and observe its transmittance reading with the meter needle again brought to zero. This value should agree within one division on the transmittance dial with the value established during calibration of the apparatus. If it does not, reset the transmittance dial to the original calibration point and zero the needle with the sensitivity control. Check the reading of the 100-ppm standard solution and if necessary readjust to a transmittance of 100. Finally, alternately use the unknown solution and the closest standard solution until readings for the unknown agree within one division on the transmittance dial and readings for the standard similarly agree with the calibration point. Record as "gross luminosity" the average of the last two transmittance readings obtained for the unknown solution. Check the blank or background correction previously obtained with the 0-ppm standard solution (No. 6).

**NOTE 7.**—As previously directed, atomize distilled water through the burner after each atomizing of a standard or sample solution.

(c) *Determination of  $Na_2O$  and  $K_2O$ .*—

(1) If the Beckman model DU instrument is to be used, determine  $Na_2O$  and  $K_2O$  as directed in Paragraph (b) except that the controls shall be set as described in Table VI. Set the wavelength dial to the point of maximum response to  $Na_2O$  or  $K_2O$  by the use of the standard calcium-free solution (No. 7).

(2) If the Perkin-Elmer instrument is to be used, determine, calculate and report  $Na_2O$  and  $K_2O$  according to ASTM Methods C 114-58 T, Section 13.<sup>2</sup>

(d) *Calculations and Reports.*—Correct the average of transmittance dial readings for background emission. Using the net luminosity values for  $MnO_3$ ,  $Na_2O$ , and  $K_2O$  in the unknown sample, read the percentage of each oxide from its respective calibration curve. Report each oxide to the nearest 0.01 per cent.



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### If only you needed a camera



From our Stuttgart factory we are now importing these *Kodak Retina Reflex Cameras*. They sell for \$215 (List) with the basic 50mm  $f/2$  *Retina Xenon-C* 6-element coated lens. Interchangeable 6-element components are available to provide a focal length of 35mm for wide angle or 80mm for telephoto work. There must be a good reason why noble optical equipment should be named after a noble gas. German engineers never do anything illogical.

With  $f/2$  and the remarkable film sensitivity now obtainable, surprisingly many pictures will be made with no auxiliary light at all. For the rest, the camera provides synchronization with all popular flashbulbs and discharge lamps at speeds up to 1/500 second.

For a happy-moments camera employed occasionally in a bat cave the coupled split-image rangefinder of the *Retina Reflex* would suffice. This camera extends itself beyond aim-and-fire applications. For technical work, as in photomicrography with the *Kodak Retina Microscope Adapter Kit, Type C*, the other and larger aspect of its character comes into play.

The rangefinder split-field is but the center of a truthful preview of the entire picture, seen upright and laterally correct. This image is formed at full lens aperture by the taking lens itself, so that even at only six inches film-to-subject distance (as with one of the *Kodak Retina Close-Up Lens Sets* instead of a microscope) the boundaries of the 24mm by 36mm picture inevitably coincide exactly on the ground glass as on the film. Since the entire

picture may be examined on its extra-fine surface, the focusing may be adjusted to favor any portion thereof without re-aiming—a simple point worth remembering when comparing cameras of this type.

*Phone a camera shop and check whether they have yet received a Kodak Retina Reflex Camera you can examine. If we had known you were interested, we would have left room here for details on the built-in photoelectric exposure meter, the mechanical linkage of shutter speed to diaphragm setting, and the way the instant before exposure it stops down to the aperture chosen.*

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Yet dithiol refused to lie down and die. Chemists have persisted in writing papers about it—"most promising colorimetric reagent for tin"; molybdenum determination in soils, metals, and rocks; precipitant for metals of the  $H_2S$  group; simultaneous determination of molybdenum and tungsten in biological materials; the zinc-dithiol complex as a reagent for arsenic and germanium; semi-microanalysis for bismuth; and on and on. There was a market there, all right.

Once we came close to doing something about it. A paper in the Indian literature, though it discussed another compound, struck us as offering a sound route to dithiol. We talked ourselves out of trying it on the grounds that dithiol had a short shelf life.

Eight more years slipped by before the break came. An inquirer so goaded us with his scoffing at our pessimism about dithiol and with his taunt about what a cinch it was that we lost our head a little and bought the right to use his

method. We don't react that way very often.

His method seems to have worked. We find ourselves in possession of a large stock bottle full of dithiol. Over the dithiol there is nitrogen, just to be surer. It is to be hoped that all who have inquired about dithiol over the past 21 years are still alive and well and interested in analytical chemistry.

Procedural abstracts on the more promising applications of dithiol are obtainable without charge from *Distillation Products Industries, Rochester 3, N. Y.* (Division of Eastman Kodak Company). Likewise obtainable is the new List No. 41 of some 3700 *Eastman Organic Chemicals*, out of which, happily, dithiol is but one.

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By reading our circulars a person can no more acquire proficiency in counterfeiting than he can in autoradiography of tissue sections, spectrography, astrophysical research, or investigations with nuclear track emulsions. Nevertheless, as long as intentions are legal, it doesn't hurt early in the game to obtain from us the essential facts about the specialized photographic materials we manufacture for these purposes.

New sheets entitled "*Kodak Sensitized Materials for Autoradiography*," "*Kodak Spectrum Analysis Plates and Films*," and "*Kodak Nuclear Track Materials*" are obtainable without charge from Eastman Kodak Company, Special Sensitized Products Division, Rochester 4, N. Y. He who asks for all three impresses us with the scope of his ambitions but strains our credulity a mite.

Prices stated include Federal Tax where applicable and are subject to change without notice.

This is another advertisement where Eastman Kodak Company probes at random for mutual interests and occasionally a little revenue from those whose work has something to do with science

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# PERSONALS...

News items concerning the activities of our members will be welcomed for inclusion in this column.

A number of ASTM members participated in the First International Skid Prevention Conference held at the University of Virginia, Charlottesville, September 8-12, 1958. President K. B. Woods, head,

School of Civil Engineering, Purdue University, presided at a session on Relationship of Road Surface Properties to Skidding. Others taking part included Honorary Member Albert T. Goldbeck, engi-

neering consultant, National Crushed Stone Association, who presided at an evening session, J. E. Gray, engineering director of the association, F. V. Hveem, materials and research engineer, California State Division of Highways, and D. D. Woodson, staff engineer of The Asphalt Inst.

William Arnott, Jr., until recently research and test engineer, Burndy Corp., Norwalk, Conn., is now director of engineering, York Research Corp., Stamford, Conn.

Julius Avins is president of Avins Industrial Products Corp., New York City. He formerly was on the chemical engineering staff of David L. Wilkoff Co., Inc.

George D. Beal, director of research at Mellon Inst., retired August 31 after 32 years of service. The author of many articles in basic chemistry, food preservatives, industrial hygiene, glass containers, scientific standards and the philosophy of research, he had taken a leading part for 25 years in the periodic revision of the U. S. Pharmacopeia. He had served on ASTM Committees D-19 on Industrial Water and C-14 on Glass and Glass Products.

John L. Butler, formerly manager, Laboratory and Inspection Service, Hardwood Plywood Inst., Atlanta, Ga., is now technical service representative, Resin Adhesives, Reichhold Chemicals, Inc., Charlotte, N. C.

Harold F. Dodge, who retired January, 1958, as quality results engineer, Bell Telephone Laboratories, after many years of service, is now on the faculty of Rutgers University, New Brunswick, N. J., as professor of applied and mathematical statistics. Mr. Dodge has been very active in ASTM Committee E-11 on Quality Control of Materials, serving as chairman since its organization in 1946 until this year.

Ray P. Dunn, formerly director of metallurgy, U. S. Reduction Co., East Chicago, Ind., has been appointed technical director, Lindberg Melting Furnace Division, Lindberg Engineering Co., Chicago, Ill.

Bram C. Feldman, formerly with the U. S. Department of the Army Corps of Engineers, Fort Belvoir, Va., is now with the Atomic Energy Commission, Washington, D. C.

After many years of constructive service, Ray A. Frye, who represented Westinghouse Electric's sustaining membership in the Society, has retired. He has been succeeded, as manager of the standards department of the Materials Engineering Departments at East Pittsburgh, by Frank V. Kupchak. This department promotes and coordinates the corporation's many activities involving materials, process and finish specifications, and engineering standards. Like his predecessor, Mr. Kupchak serves on numerous committees and is affiliated with the Standards Engineers Society. The representative of the Westinghouse sustaining membership will be Stanley A. Rosecrans, assistant manager, Materials Engineering Depart-

(Continued on page 70)

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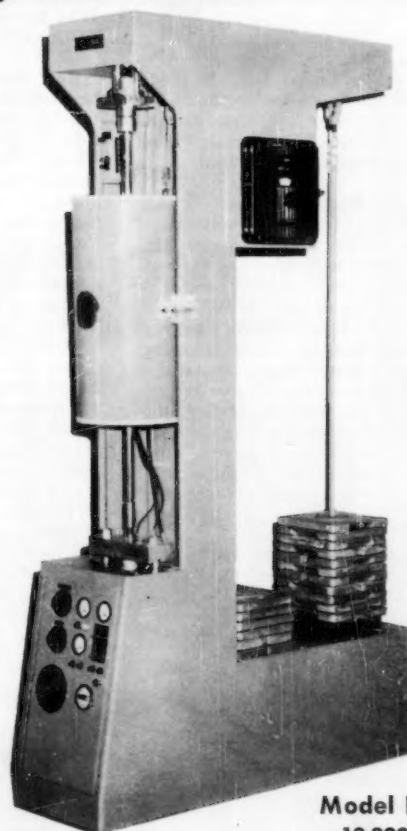
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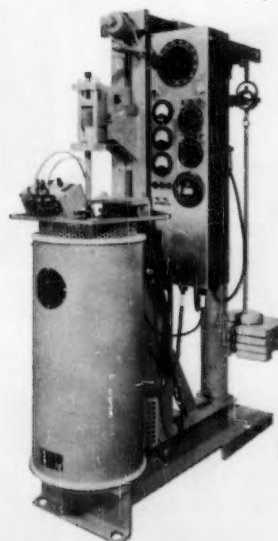
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ASTM BULLETIN

**Budd**



## Personals

(Continued from page 68)

ments, who has been appointed to the ASTM Administrative Committee on Standards.

**LeRoy A. Grotto**, formerly on the research staff, Mack Trucks, Inc., Plainfield, N. J., is now development engineer, International Harvester Co., Construction Equipment Division, Development and Research Laboratory, Melrose Park, Ill.

**Herbert H. Hatch**, president, Hatch Textile Research, Inc., is celebrating his fiftieth year in the textile industry. In September, 1908, he entered his father's public textile adjusting business located in the old Wrigley Building at 192 W. Broadway, New York City. In 1926, recognizing the need for higher standards of testing he established a textile testing laboratory. For the past 19 years he has been located at 25 E. 26th St., New York City, the laboratory handling many phases of textile testing and specializing in construction, wearability, "wash and wear," and quality control tests for all textile users and distributors.

**Oliver M. Hayden**, who recently retired as manager of technical section, Rubber Chemicals Division, Organic Chemicals Dept., E. I. du Pont de Nemours and Co.,

Inc., Wilmington, Del., was elected to honorary membership in ASTM Committee D-11 on Rubber and Rubber-Like Materials, recognizing long-time service and valued contributions. Mr. Hayden, who had served as chairman of Committee D-11 for six years, was honored by the Society with an Award of Merit in 1954.

**Rear Admiral Leon J. Jacobi** (USNR) has been elected national president of the Reserve Officers Association 1958-1959. Admiral Jacobi's civilian position is inspection and standards engineer of the Detroit Edison Company. He has been a member of ASTM since 1946 and is active in the work of Committees A-5 on Corrosion of Iron and Steel and D-7 on Wood. He is also a member of the Administrative Committee on Standards and the Detroit District Council.

**Jerome J. Kanter**, directing engineer, Crane Co., Chicago, was among nine ASME members recently honored by election to the grade of Fellow of The American Society of Mechanical Engineers. An acknowledged authority on the effect of temperature on the properties of metals, Mr. Kanter has been very active also in ASTM for many years. For more than 20 years he has been a member of the Joint ASTM-ASME Committee on Effect of Temperature on the Properties of Metals. Currently he serves as vice-chairman of this joint committee, and as chairman of

its Test Methods Panel. He is now chairman of Committee A-1 on Steel and has long been active in this committee. In 1952 he was among the recipients of the ASTM Award of Merit in recognition of his "consistent and constructive work in standards and research over many years."

**Raymond F. Ledford**, formerly sales manager, Industrial Filter and Pump Manufacturing Co., Chicago, is now president, Chemray Corp., Riverside, Ill.

**Arthur A. Levison** recently retired as vice-president and general sales manager, Blaw-Knox Equipment Division, Blaw-Knox Co., Pittsburgh, Pa. Mr. Levison has been a member of ASTM for more than 30 years, and active in Committee C-9 on Concrete and Concrete Aggregates for the entire period. He resides at 1607 Shady Ave., Pittsburgh.

**James J. Lingane**, chairman, Chemistry Dept., Harvard University, received the Fisher Award of the American Chemical Society. The Award carries a prize of \$1000.

**Wheeler G. Lovell** was promoted to research adviser in charge of research planning at Ethyl Corp.'s Detroit research laboratories.

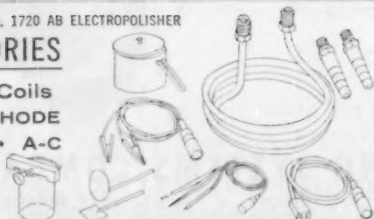
(Continued on page 72)



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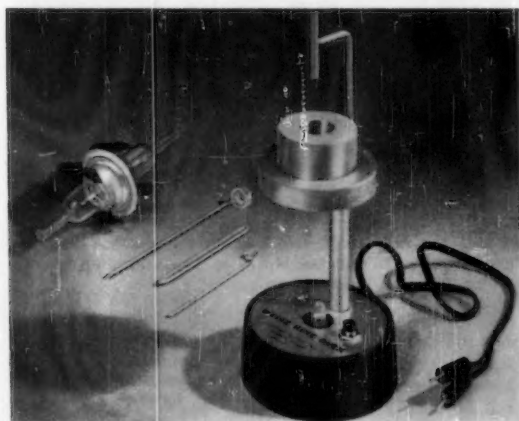
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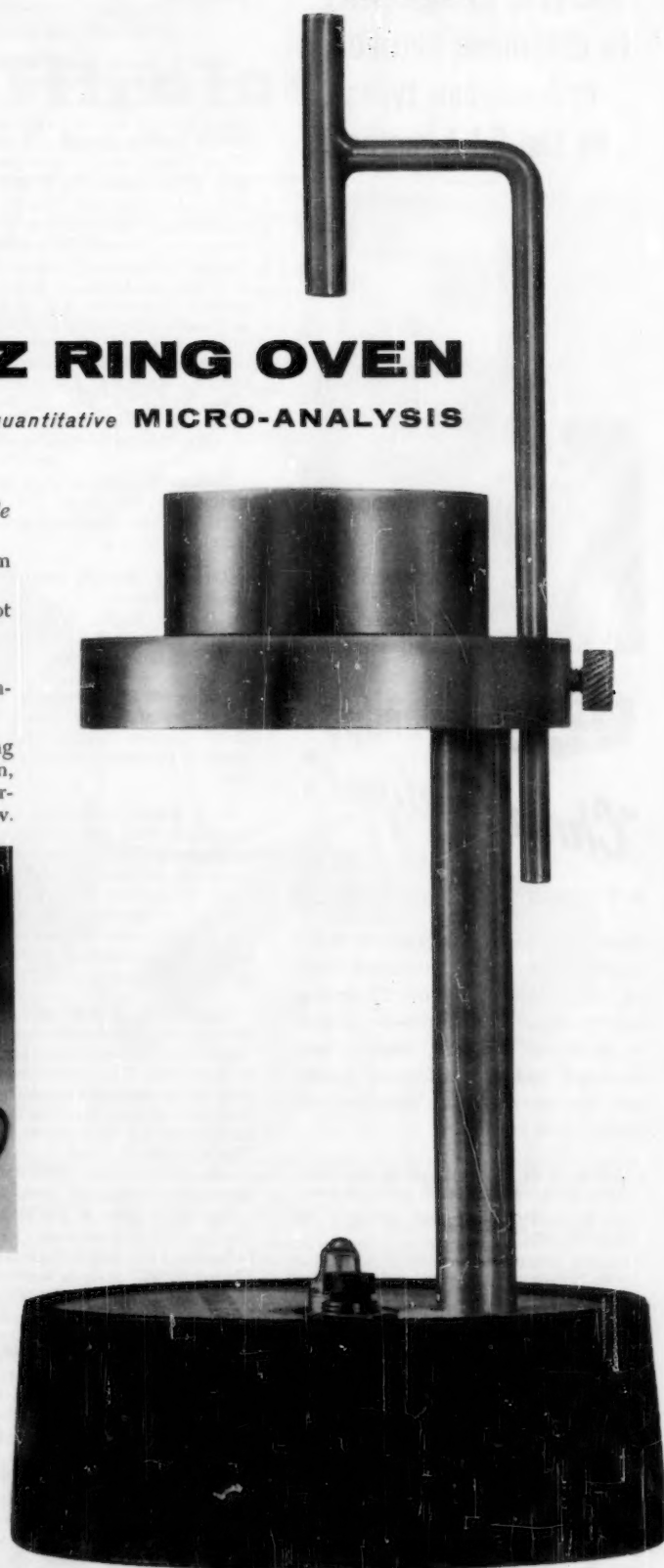
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October 1958

ASTM BULLETIN

71

actual size



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CIRCLE 1035 ON READER SERVICE CARD

### Personals

(Continued from page 70)

Emil A. Lucas, long-time member of the Society, and vice-president and works manager, Molybdenum Corp., Washington, Pa., delivered the main address at the Ninety-second Founders Convocation of Thiel College earlier this year when the ground was broken for Thiel's new science hall. His topic was "Thiel's Aspirations in Science." He is chairman of the Committee on Business Management of the College's Board of Trustees. Dr. Lucas is active in ASTM technical work, especially Committee A-9 on Ferro-Alloys, and heads its Specifications Subcommittee.

Oscar O. Miller, supervisor of steel section, Research Laboratory, International Nickel Co., Inc., Bayonne, N. J., is 1958-1959 president of The Technical Societies Council of New Jersey.

George H. Obear, formerly with J. E. Greiner Co., Baltimore, Md., is chief soils engineer, Ewin Engineering Corp., Washington, D. C.

Arthur J. Raymo, until recently with General Electric Co., Atomic Power Equipment Dept., San Jose Calif., is factory manager, Baldwin-Lima Hamilton Corp., Philadelphia, Pa.

James M. Rice, formerly with the National Crushed Stone Assn., Washington, D. C., is now director, Natural Rubber Bureau, Research Laboratory, Arlington, Va.

S. J. Rosch, consulting cable engineer, Anaconda Wire and Cable Co., Hastings-on-Hudson, N. Y., has been elected an honorary member of ASTM Committee D-11 on Rubber and Rubber-like Materials. A member of the committee for the past 25 years, he has been especially active in the work of the subcommittee on insulated wire and cable.

Charles R. Stock, national director, who in June transferred his activities with American Cyanamid from Stamford, Conn., to Pensacola, Fla., where the Santa Rosa plant of the company is getting into operation, has advised that his family and he have made the adjustment rather easily. This Cyanamid Plant is to manufacture Creslan acrylic fiber. It is about 16 miles northeast of Pensacola, in the same area as a number of other ASTM company members, including Armstrong Cork, Newport Industries, St. Regis Paper, and others. The plant will use raw materials, mainly acrylonitrile from the New Orleans Plant. Mr. Stock's responsibilities cover supervision and direction of the Chemical Control Laboratories, the Physical Testing Laboratories, the Dye Laboratories, Microscopy, and the Textile Processing Laboratories. Their functions are to control the production operations as close to predetermined optima as possible, to evaluate the quality of the resulting fiber and to grade or reject it for sale, to learn the sources of defects leading to downgrading or rejection and to take steps to eliminate the causes, and to review continually for correlations between any of the properties

related to use, physical behavior, textile processing, dyeing, and the chemistry leading up to and including polymerization. Mr. Stock's address is Post Office Drawer 1826, Pensacola, Fla.

Sd. Tjokronolo, until recently chief, Government Central Purchasing Office, Djakarta, Indonesia has been assigned to a new post in New York City as chief, Indonesian Supply Mission.

L. G. Toye retired September 1, after 39½ years' service with General Electric Co., Erie (Pa.) Works. He had been supervisor of the Metallurgy Laboratory, Locomotive and Car Equipment Department. Mr. Toye had represented the company on a number of the metals committees.

Earl E. Wagner, formerly with Hoover Ball and Bearing Co., Ann Arbor, Mich., has accepted a position as customer contact engineer, Michigan Seamless Tube Co., South Lyon, Mich.

## DEATHS...

R. R. Bowser, director, Bowser-Morner Testing Laboratories, Dayton, Ohio, died July 24, 1958. Mr. Bowser had represented the company membership since 1929. He served on the Ohio Valley District Council from 1949 to 1951 and as a member of the organizing committee of that district.

Dana James Demorest, professor of metallurgical engineering, College of Engineering, The Ohio State University, Columbus, died June 30, 1958. A member of the Society for 41 years, he had served on Committees A-7 on Malleable-Iron Castings and D-5 on Coal and Coke. Professor Demorest had been connected with Ohio State Department of Metallurgy since 1907.

Frederick A. McCoy, chief metallurgist, Sheffield Steel, Division of Armco Steel Corp., Kansas City, Mo., died August 25, 1958. He had represented the company membership since 1944, serving on Committee A-1 on Steel.

C. A. Neusbaum, laboratory director, Standard Inspection Laboratory, Esso Research Center, Linden, N. J., died August 29, 1958. Representative of the sustaining membership of the center for many years, Dr. Neusbaum had a long record of valued service (dating from 1931) in Committee D-2 on Petroleum Products and Lubricants. He was a member of the D-2 Advisory Committee for 14 years, and had served on many of the D-2 technical committees and research divisions. For a number of years he headed Research Divisions II on Measurement and Sampling, and V on Analysis of Fuels. Dr. Neusbaum is survived by his wife Connie who lives in Sparta, N. J. (Box 412).

L. E. Pitzer, chemical engineer, United States Steel Corp., Pittsburgh, Pa., died May 2, 1958. A member of the Society

(Continued on page 74)



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6. Directly applicable to all types of redox, acid-base, precipitation and complexation titration systems.
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based on the original work of H. V. Malmstadt, (Anal. Chem., 26, 1348[1954]).

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For use in all titrations usually classified as potentiometric. Consisting fundamentally of a mixing and delivery unit with associated supports and an electronic control circuit.

The control unit contains the detecting, amplifying, differentiating and relaying circuits with a simple operational panel. At the rear of the control unit are a stirring motor switch for manual use, a fuse, connecting outlets to the valve unit, connecting outlet to the power supply, and a recorder outlet permitting, for research purposes, the recording of the second derivative curve with automatic cutoff.

The delivery unit contains a valve mechanism with an adjustable rate controller, an open ended motor driven chuck for the stirrer, stirring motor and electrode holders. The valve mechanism,

mounted on a support with swinging beaker platform and a clamp for the titrating burette, is designed to accommodate any form of burette with or without stopcocks, so that it may apply to any type of titrating installation.

Dimensions of control unit: height, 7 $\frac{1}{4}$  inches; width, 10 inches; depth, 9 inches; power consumption, 100 watts; total shipping weight, 35 lbs.

Complete, including: control unit; stirring valve unit; support stand with beaker platform and burette clamp; one S-30490 calomel electrode; one each S-30440 electrode, platinum and platinum rhodium; one S-29695 delivery tip with internal platinum electrode, one S-76667 stirring rod; one S-76668 large stirring rod; connecting cable for recorder; and connecting cord and plug for standard outlets. Without beakers or burettes. For operation from 115 vol, 60 cycle A.C. circuits..... **\$450.00**

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FOR FURTHER INFORMATION CIRCLE 1036 ON READER SERVICE CARD

## Deaths

(Continued from page 72)

since 1946, Mr. Pitzer had served on Committees A-9 on Ferro-Alloys, D-19 on Industrial Water, E-2 on Emission Spectroscopy, and E-3 on Chemical Analysis of Metals.

**O. Ben Schultz**, retired chief metallurgist and manager of inspection, Construction Equipment Division, Baldwin-Lima-Hamilton Corp., Lima, Ohio, died March, 1958. Mr. Schultz had been connected with the company 37 years, having started as chief chemist when the Division was formerly Lima Locomotive Works. He had been very active for many years prior to his retirement in 1954 in Committees A-1 on Steel, A-2 on Wrought Iron, and A-3 on Cast Iron. Mr. and Mrs. Schultz both had attended many ASTM annual meetings. Mrs. Schultz resides at 1537 Oakland Parkway, Lima, Ohio.

**A. E. Stoddard**, assistant engineer of materials and tests, Iowa State Highway Commission, Ames, died August 30, 1958. A member of the Society since 1922, Mr. Stoddard had served for many years on Committees C-9 on Concrete and Concrete Aggregates, D-4 on Road and Paving Materials, and D-18 on Soils for Engineering Purposes.

## NEW MEMBERS.....

The following 55 members were elected from August 14 to September 11, 1958, making the total membership 9515..... Welcome to ASTM

Note—Names are arranged alphabetically—Company members first then individuals—Your ASTM Year Book shows the areas covered by the respective Districts

**CENTRAL NEW YORK DISTRICT**  
**Fleischmann, Walter L.**, consulting engineer, Knolls Atomic Power Laboratory, General Electric Co., Schenectady 1, N. Y.  
**Johnson, Raymond P.**, assistant manager, Petroleum Loss Control, Socony Mobil Oil Co., Inc., 150 E. Forty-second St., New York 17, N. Y. For mail: Box 76, New Baltimore, N. Y.  
**Mapes, Robert W.**, test development group leader, Behr-Manning Co., Troy, N. Y. For mail: 14 Brookwood Dr., Latham, N. Y.

**CHICAGO DISTRICT**  
**Chrysafopoulos, Nicholas**, assistant professor of civil engineering, University of Illinois, 208 Talbot Laboratory, Urbana, Ill.  
**Oleson, Calvin C.**, principal research engineer, Portland Cement Assn., 5420 Old Orchard Rd., Skokie, Ill.  
**Olson, Eugene O.**, chief engineer, Delavan Manufacturing Co., 811 Fourth St., West Des Moines, Iowa.

**Olson, Roy E.**, instructor, Department of Civil Engineering, University of Illinois, 206 Talbot Laboratory, Urbana, Ill. [A]\*  
**Reisenbiger, A. F.**, president, Erie Casein Dryers, Box 55, Erie, Ill.

**CLEVELAND DISTRICT**  
**Glasic Corp., The, Roger S. Jackson**, chief engineer, 4321 Glenridge Rd., Cleveland 21, Ohio.  
**Baird, John H.**, sales manager, Special Products, The Lubrizol Corp., Cleveland 17, Ohio. For mail: 2914 Attleboro Rd., Shaker Heights, Ohio.  
**Craig, Willis G.**, director, Chemical Products Development and Application The Lubrizol Corp., Cleveland 17, Ohio. For mail: R. D. 2, Metcalf Rd., Waite Hill, Willoughby, Ohio.

**DETROIT DISTRICT**  
**McKenzie, T. Curtis**, president, Klem Chemicals, Inc., 14401 Lanson St., Dearborn, Mich.  
**Payne, Edwin B.**, supervisor, Physical and Chemical Testing, Ford Motor Co., Chemical Products Plant, 151 Lafayette, Mt. Clemens, Mich.

**NEW ENGLAND DISTRICT**  
**Murphy, Robert J.**, head, Analytical Laboratory, M & C Nuclear, Inc., Attleboro, Mass.

**NEW YORK DISTRICT**  
**Campagna, Charles**, president, Spectranome Plating Co., Inc., 330 W. Thirteenth St., New York 14, N. Y.  
**Dater, Arnold H.**, chief chemist, Ultra Chemical Works, Inc., 2 Wood St., Paterson, N. J.  
**Marlowe, Sherwood V.**, soils inspector, E. Lionel Pavlo, Consulting Engineers, 642 Fifth Ave., New York, N. Y. For mail: 152 County Ave., Secaucus, N. J. [A]  
**Marron, Albert J.**, corrosion engineer, the International Nickel Co., Inc., 67 Wall St., New York 5, N. Y. [A]  
**Roos, Sigmund**, consulting engineer, 144 E. Thirtieth St., New York 16, N. Y.  
**Uffner, Melville W.**, head, Rubber and Plastics Division, Thompson, Weinman and Co., 92 Greenwood Ave., Montclair, N. J.

**NORTHERN CALIFORNIA DISTRICT**  
**Bunting, Frederick B.**, section head, Bridge Section, Porter, Urquhart, McCreary & O'Brien, 1140 Howard St., San Francisco 3, Calif. For mail: 389 Guerrero St., San Francisco 3, Calif.

**OHIO VALLEY DISTRICT**  
**Lipsitt, Harry A.**, project scientist, Wright Air Development Center, Wright Patterson Air Force Base, Ohio. For mail: 338 Chaucer Rd., Dayton 31, Ohio. [A]

**PHILADELPHIA DISTRICT**  
**Stressteel Corp., J. N. Hicks**, vice-president, 221 Conyngham Ave., Wilkes-Barre, Pa.  
**Clark, George A.**, supervisor, Engineering and Inspection Dept., United States Testing Co., Inc., 3925 M St., Philadelphia 24, Pa. For mail: 2003 Allen St., Allentown, Pa.

(Continued on page 76)

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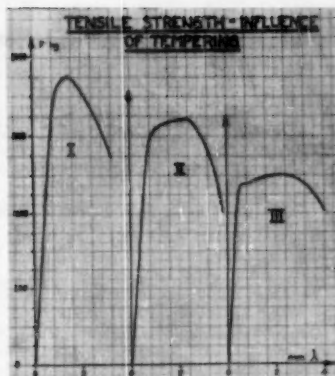
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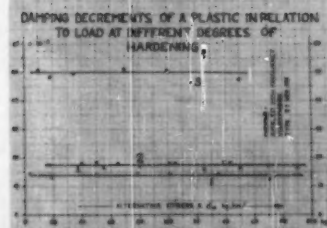
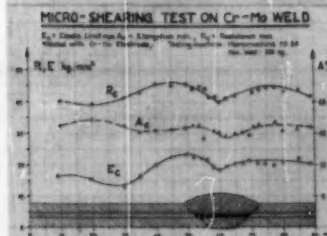
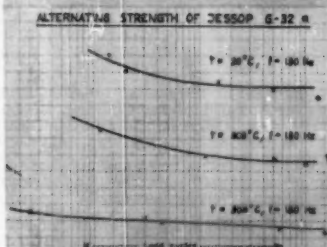
Weather-Ometer® Fade-Ometer® Launder-Ometer® Random Tumble Pilling Tester Scorch Tester Accelerator®

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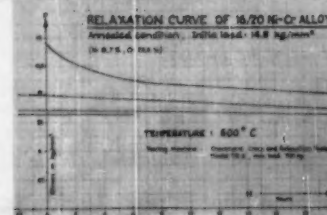


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## New Members

(Continued from page 74)

**Gates, Albert M.**, senior engineer, Research and Development Dept., Philadelphia Electric Co., 1000 Chestnut St., Philadelphia 5, Pa. For mail: 6113 McCallum St., Philadelphia 44, Pa.

**Harter, John William**, laboratory technician, United Testing Laboratories, Inc., Box 101 S. Broad St., Trenton 20, N. J.

**Spencer, L. Paul**, chief chemist, Walker Brothers, End Street and R. R., Conshohocken, Pa.

### PITTSBURGH DISTRICT

**Syntrom, Co.**, Paul C. Zanotto, assistant manager, Parts Handling Equipment Dept., Homer City, Pa.

**Faries, D. T.**, chief engineer, Bessemer & Lake Erie Railroad Co., Greenville, Pa.

**Williams, Robert N.**, director of research, Reliance Steel Products, McKeesport, Pa. For mail: 3700 Walnut, McKeesport, Pa.

### ST LOUIS DISTRICT

**Canham, Walter G.**, manager, Standards Section, Monsanto Chemical Co., Lindbergh and Olive Street Rd., St. Louis 24, Mo.

**Stonebraker, Hal**, architect, 7134 N. Eightieth St., Overland Park, Kans.

### SOUTHEAST DISTRICT

**Culbertson, Lowell E., Jr.**, resident engineer, Bowater's Southern Paper Corp., Calhoun, Tenn.

**Frederickson, Charles**, technical director, Southern Electrical Co., Division of Olin Mathieson Chemical Corp., Box 989, Chattanooga 5, Tenn.

**Griffin, William R.**, technical service engineer, Edgar Plastic Kaolin Co., Edgar, Fla.

### SOUTHERN CALIFORNIA DISTRICT

**Atomics International**, Albert P. Bradley, supervisor, Library, Box 309, Canoga Park, Calif.

**Smith, Clarence R.**, design specialist, Convair, A Division of General Dynamics Corp., San Diego 12, Calif. For mail: 1807 Beryl St., San Diego 9, Calif.

### SOUTHWEST DISTRICT

**Chaumont, Louis F.**, resident inspector, Pittsburgh Testing Laboratory, Box 1861, Oil Center Station, Lafayette, La.

### WASHINGTON (D. C.) DISTRICT

**Heath, George A.**, laboratory manager, Collins & Aikman Corp., Cavel, N. C. For mail: R. F. D. 1, Chub Lake St., Roxboro, N. C.

**Howie, Thomas L., Jr.**, chemist, Sidney Blumenthal and Co., Inc., Carr St., Rocky Mount, N. C.

**Smith, Preston C.**, chief, Soils Branch, Division of Physical Research, U. S. Bureau of Public Roads, Washington 25, D. C. For mail: 420 Tyler Pl., Alexandria, Va.

**Wieczorek, T. F.**, sales manager, Eastern Products Corp., 1601 Wicomico St., Baltimore 30, Md.

### WESTERN NEW YORK-ONTARIO DISTRICT

**Shifflett, E. W.**, Engineering Dept., Leland Electric Canada, Ltd., Crimea St., Guelph, Ont., Canada.

### OUTSIDE ESTABLISHED DISTRICTS

**Wah Chang Corp.**, J. H. McClain, manager of manufacturing, Box 366, Albany, Ore.

**Miles, M. J.**, chief chemist, Titanium Metal Corporation of America, Box 2128, Henderson, Nev. For mail: Box 168, Henderson, Nev.

**Wright, Howard W.**, test requirements planner, Boeing Airplane Co., 7755 E. Marginal Way, Seattle 8, Wash. For mail: 7310 Twentieth Ave., N. E., Seattle 15, Wash.

### OTHER THAN U. S. POSSESSIONS

**General Motors do Brasil S/A**, E. M. Kennedy, chief engineer, Caixa Postal 8200, Sao Paulo, Brazil.

**Baughan, John J.**, project engineer, Argo Block Co., Ltd., Cooksville, Ont., Canada.

**Daglio, Ernesto**, owner-president, Fina Calle Gerardo Barrios, San Salvador, E. Salvador.

**Det norske Veritas**, Library, Rådhusgt 25 Oslo, Norway.

**Ermyny-Imery, J. S.**, civil engineer, Construcciones ERIMECA, Edif. Unidad Tecnica del Este, A. V., Libertador, 7° Piso, Oficinas 16 y 17, Caracas, Venezuela. For mail: Apartado Postal 6441, Caracas, Venezuela.

**Hamid Ali, Syed Mohammed**, chemist, Technical Dept., Standard Vacuum Oil Co., Karachi, Pakistan. For mail: Ethel Villa, Catholic Colony No. 1, Karachi-5, Pakistan.

**Philippines, Bureau of Public Highways**, Technical Library, Columna S. Lizardo, Bureau Librarian, Second St. Port Area, Manila, Philippines.

**Romeo Salsa, Albert**, chief lubrication technician, Orinoco Mining Co., Apartado 3, Ciudad Bolivar, Venezuela.

**Wintermark, Herman**, assistant chief, Det norske Veritas, Box 82, Oslo, Norway.

\*[A] denotes Associate Member.

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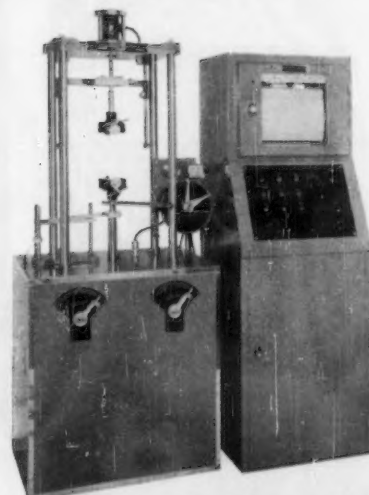
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## OTHER SOCIETIES' EVENTS

- November 3-8—8th Plenary Meeting, ISO/TC 61 on Plastics, Washington, D. C.
- November 5-7—Society of Rheology, Annual Meeting, Franklin Institute, Philadelphia, Pa.
- November 6-7—American Society for Quality Control, 13th Midwest Quality Control Conference, Kansas City, Mo.
- November 10-12—Steel Founders' Society of America, 13th Technical and Operating Conference, Carter Hotel, Cleveland, Ohio.
- November 10-12—Structural Clay Products Institute, Annual Convention, Shamrock Hilton Hotel, Houston, Tex.
- November 10-12—Atomic Industrial Forum, Annual Nuclear Industry Conference, Shoreham Hotel, Washington, D. C.
- November 10-13—American Petroleum Institute, Annual Meeting, Conrad Hilton, Palmer House and Congress Hotels, Chicago, Ill.
- November 10-13—National Electrical Manufacturers Assn., Annual Meeting, Traymore Hotel, Atlantic City, N. J.
- November 12-14—Society for Experimental Stress Analysis, Annual Meeting, Hotel Sheraton-Ten Eyck, Albany, N. Y.
- November 12-15—Society of Naval Architects and Marine Engineers, Annual Meeting, Waldorf Astoria Hotel, New York, N. Y.
- November 17-20—Conference on Magnetism and Magnetic Materials; joint sponsors: American Institute of Electrical Engineers, American Physical Society, Institute of Radio Engineers, Metallurgical Society of AIME, and Office of Naval Research; Sheraton Hotel, Philadelphia, Pa.
- November 17-21—Society of the Plastics Industry, Inc., 8th National Plastics Exposition, International Amphitheater; and Annual Conference, Morrison Hotel, Chicago, Ill.
- November 18-20—The National Conference on Air Pollution; Department of Health, Education, and Welfare, Public Health Service; Sheraton-Park Hotel, Washington, D. C.
- November 18-20—American Standards Assn., 9th National Conference on Standards, The Hotel Roosevelt, New York, N. Y.
- November 20-21—National Foundry Assn., Annual Meeting, Drake Hotel, Chicago, Ill.
- November 20-22—Acoustical Society of America, Fall Meeting, Congress Hotel, Chicago, Ill.
- November 25—Manufacturing Chemists' Assn., 8th Semiannual Meeting and Winter Conference, Hotel Statler, New York, N. Y.
- November 30-December 5—The American Society of Mechanical Engineers, Annual Meeting, Statler and Sheraton-McAlpin Hotels, New York, N. Y.
- December 1-3—American Society of Refrigerating Engineers, Semiannual Meeting, Hotel Roosevelt, New Orleans, La.
- December 1-4—National Warm Air Heating and Air Conditioning Assn., Annual Convention, Cleveland, Ohio.
- December 2-4—Electronic Industries Assn., 3rd Conference on Reliable Electrical Connections, Statler-Hilton Hotel, Dallas, Tex.
- December 7-10—American Institute of Chemical Engineers, Annual Meeting, Netherland Plaza, Cincinnati, Ohio.
- December 8-10—American Nuclear Society, Winter Meeting, Sheraton-Cadillac Hotel, Detroit, Mich.
- December 17-19—American Society of Agricultural Engineers, Winter Meeting, Palmer House, Chicago, Ill.
- December 26-31—American Association for the Advancement of Science, Annual Meeting, Sheraton-Park Hotel, Washington, D. C.
- December 27-30—American Statistical Assn., Congress Hotel, Chicago, Ill.

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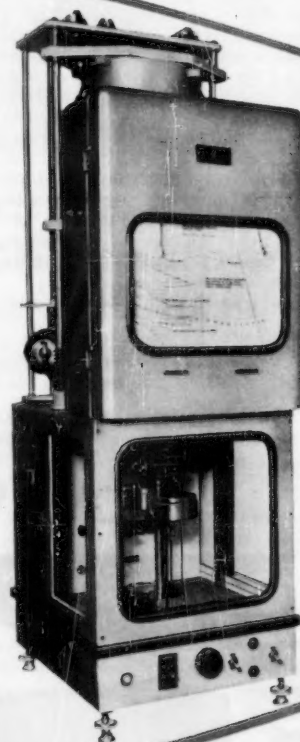
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## The Bookshelf

### Electrostatics in the Petroleum Industry

Edited by A. Klinkenberg and J. L. van der Minne, Elsevier Publishing Co., New York, N. Y., 191 pp., \$8. (1958)

THIS PUBLICATION records investigations of the accidental ignition of petroleum vapors by discharge of static electricity and methods for reducing its occurrence, subjects which have long been of interest from the standpoint of fire and explosion prevention.

Results are presented of laboratory-scale determinations of the electrical charges generated by flowing liquid hydrocarbons, both with and without additives for increasing the electrical conductivity of the liquid and thus reduce accumulation of the static electrical charges. A method for determining the electrical conductivity of petroleum liquids is described.

Of greatest interest from a practical standpoint are accounts of full-scale tests using actual refinery tanks, pipe lines, and associated equipment. Ignition of flammable vapor-air mixtures

within these refinery tanks by discharge of static electricity generated on pumping various petroleum liquids into the tanks under controlled conditions occurred in several of these tests. Where certain additives were present in the petroleum liquids, no explosion occurred in the tests. Earthing of all equipment is, however, still considered necessary.

The physical basis and the factors influencing the generation and accumulation of static electrical charges in oil storage tanks, pipe lines, and other equipment are discussed theoretically. The conditions under which hazardous spark or corona discharges of static electricity occur are likewise discussed, and the mechanism by which they ignite flammable vapor-air mixtures. Mathematical treatment of these features is included in many instances.

An extensive bibliography on static electricity is included, together with references to technical literature relating to the ignition of flammable gases or vapors by discharge of static electricity.

This publication should be of special

interest to fire prevention engineers and others concerned with the safe handling and storage of volatile petroleum liquids.

A. F. MATSON

### Supplementary List of Publications of the National Bureau of Standards, July 1, 1947, to June 30, 1957.

National Bureau of Standards Supplement to Circular 460, U. S. Government Printing Office, Washington 25, D. C., 373 pp., \$1.50.

THE National Bureau of Standards publications listed in this Supplement to Circular 460 include Applied Mathematics Series, Building Materials and Structures Reports, Circulars, Handbooks, Research Papers, and Miscellaneous Publications. The catalog gives a brief abstract of each paper, and also contains author and subject indexes. The procedures for purchasing the publications are outlined, and a list of depository libraries throughout the United States is included.

(Continued on page 80)

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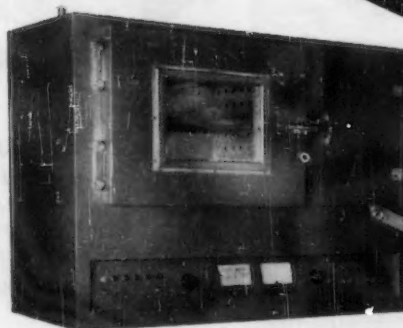
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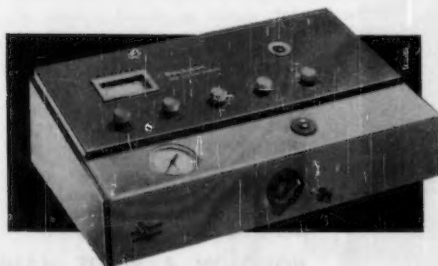
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ASTM BULLETIN

## (Continued from page 78)

Rubber Manufacturers Assn., Inc., 444  
Madison Ave., New York 22, N. Y.  
(1958), 8½ by 11 in., 52 pp., \$1.

To the extent that the handbook may help customers avoid "overspecification" in terms of such things as dimensional tolerances, trim, flash extension, appearance, load deflection, strength of bond or splices, etc., the handbook could operate to help them reduce costs.

Edited by Phillip Morgan, *Philosophical Library*, New York, N. Y., second edition (1957); 276 pp. plus 50 plates; \$15.

This book will be of particular interest and help to workers not entirely familiar with the field of glass reinforced plastics. Chapters on glass fibers and resins may help to clear up questions regarding variations and types of fiberglass reinforced products. Although most of the material in this

Perhaps the chapter of greatest interest is that on a continuous resin injection system for glass fiber moulding. This method involves forcing a resin under pressure and with automatic controls into a molding cavity pre-filled with glass fiber. Data and drawings of this system indicate it may be the first method to enable glass reinforced plastics to break loose from the hand layup techniques normally used to machine mass production.

MURRAY C. SLONE

(Continued on page 82)

## D412(51T)



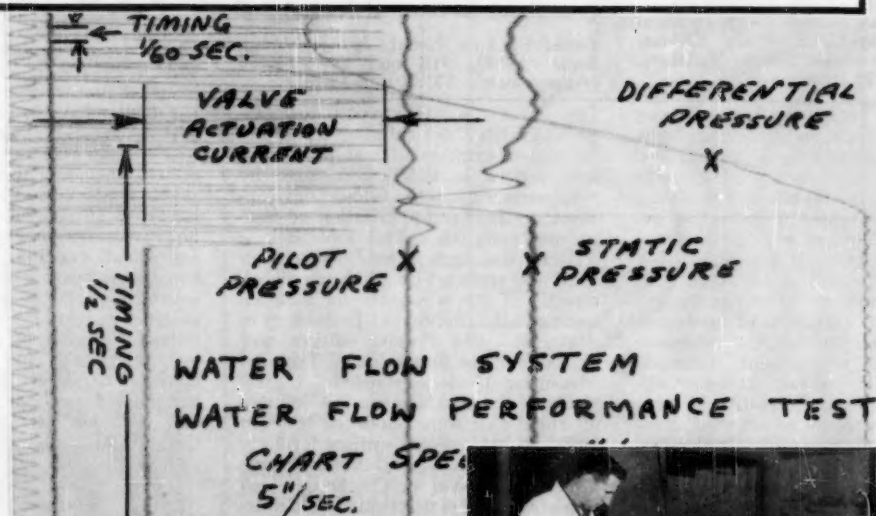
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FOR FURTHER INFORMATION CIRCLE 1043 ON READER SERVICE CARD

## This is a record of a missile component



Visicorder Record—actual size

Wyle Laboratories in El Segundo, California, have used a battery of four Visicorder consoles like the one shown (right) to run a series of tests on a vital missile component. In the Wyle test project, the unique Visicorder consoles are easy to operate. Most parameters are low frequency, requiring response on the order of 5 to 60 cycles.

The two calibrator control panels in each Visicorder console accommodate 10 plug-in balance and matching units—designed to match tachometer generators, pressure transducers, thermocouples, expanded-scale voltmeters, etc., to the Heiland galvanometers.

Dick Johnson, Instrumentation Branch Head at Wyle, says, "This system, I feel, is one of the most efficient instrumentation consoles in operation. Set-up and calibration time has been reduced by the use of Visicorders by approximately 50%. This is due to the simplicity of operation and trouble-free performance. There are no inking pens to clean, high-gain amplifier maintenance, and so on, and we can also use these consoles together to form systems of more than six channels."



Tom Jackson, Wyle engineer, examines Visicorder record

The HONEYWELL VISICORDER is the first high-frequency, high-sensitivity direct recording oscillograph. In laboratories and in the field everywhere, instantly-readable Visicorder records are pointing the way to new advances in product design, rocketry, computing, control, nucleonics ... in any field where high speed variables are under study.

To record high frequency variables—and monitor them as they are recorded—use the Visicorder Oscillograph. Call your nearest Minneapolis-Honeywell Industrial Sales Office for a demonstration.

# Honeywell



Industrial Products Group

Reference Data: Write for Visicorder Bulletin

Minneapolis-Honeywell Regulator Co., Industrial Products Group, Heiland Division, 5200 E. Evans Ave., Denver 22, Colorado

FOR FURTHER INFORMATION CIRCLE 1044 ON READER SERVICE CARD

October 1958

ASTM BULLETIN

81



## The Bookshelf

(Continued from page 80)

### Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to Alkali-Aggregate Reaction.

Technical Report Number 6-481; Technical Liaison Branch, U. S. Army Engineer Waterways Experiment Station, Vicksburg, Miss. (1958); 75 cents.

THIS report concerns twenty materials, representing eight different classes of mineral admixtures, which were evaluated, using both chemical and mortar-bar test methods, for their effectiveness in preventing excess expansion of concrete due to alkali-aggregate reaction. It was found that the chemical tests cannot be used with reliance to evaluate effectiveness, and the mortar-bar test method needs improvement to increase its precision. Each of the replacement materials evaluated will prevent excessive expansion if a sufficient quantity is used. Correlations were found between effectiveness and fineness, dissolved silica and percentage of alkali retained by reaction product. The eight classes of mineral admixtures represented in the

tests were: granulated blast-furnace slags, natural cements, fly ashes, natural volcanic glasses, calcined opaline shales, uncalcined diatomite, uncalcined quartz, and synthetic pure silica glass.

### Handbuch der Werkstoffprüfung (Handbook for Material Testing)

Edited by Erich Siebel, Springer-Verlag, Berlin (1958); 916 pp., DM 148.50 (Approximately \$35.50), in German.

A "HANDBOOK for Material Testing," which offers a comprehensive description of test procedures and devices, is widely recognized by many who work in the fields of development, testing, and application of engineering materials. This handbook is a German language publication compiled and arranged by Dr. Erich Siebel, director of the laboratory for material testing at the Institute of Technology in Stuttgart. The revised edition now available of the first volume—Test and Measuring Devices—concludes the revision of the book's entire five volumes of which four were published between 1939 and 1944. Some sections formerly included in the second volume have been incorporated in the first, which consists mainly of descriptions and illus-

trations of measuring and test devices and their modes of operation.

One of the many merits of this reliable reference work is the detailed coverage of the multitude of innovations that have appeared during the last decade in nondestructive testing and in the design of measuring and test devices brought about by the rapidly growing industrial application of electronics and low-voltage techniques. Older designs contained in the first edition of the handbook have been included in the second edition only if the devices have to be regarded as prototypes for special testing tasks. The text is again divided according to types of test, including testing machines for steady loads, impact loads, and oscillating loads, hardness testing, a general but detailed investigation of testing equipment, methods and devices for the measurement of deformation, optical and X-ray stress measurements, non-destructive testing with ultrasonic and inductive procedures, and metallographical, chemical, and spectrochemical tests. The different sections of the handbook have been prepared and reviewed by leading experts in each particular field of material testing.

GGD

(Continued on page 92)

# THE METTLER H-5 MULTI-PURPOSE BALANCE

is the perfect tool for CONTROL WEIGHINGS  
ROUTINE ANALYSES

#### VITAL STATISTICS:

**CAPACITY: 160 G**  
**RANGE OF OPTICAL SCALE: 1 G**  
**ONE SCALE DIVISION IS 10 MG**  
**EASY MICROMETER READING TO 0.2 MG**  
**And remember: The set of weights is built into the balance. No loose weights are used.**

**METTLER manufactures a very complete line of scales and balances. Write today for our file with full details.**



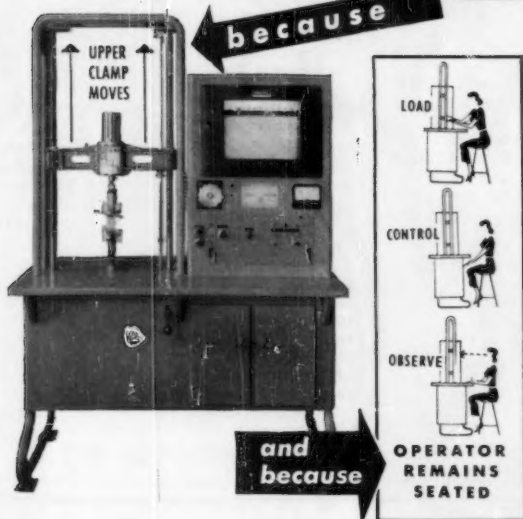
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your head to observe  
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sile Tester for High- and Low-  
Elongation Materials



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highest precision and new economy.

TESTS PRACTICALLY ANY MATERIAL IN  
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4275-R3 with  
4275-F6  
and 4275-Z.

STANDARD MODEL NO. 3

## WILEY LABORATORY MILL

- on new *Portable Stand*  
and with *Spillage Tray*

For preparation, with minimal loss of moisture from heating, of a wide variety of materials for analysis. Principal advantages of new model: harder cutting edges permitting wider range of materials including Teflon, polyethylene resins, titanium scrap, etc.; quieter operation; and baked gray enamel and chromium plated finish, etc.

As in earlier model, four hardened steel knives on revolving shaft work with shearing action against six knives bolted into frame. Shearing action of cutting edges, between which there is always clearance, minimizes loss of moisture, avoids temperature rise, liquefaction, contamination, etc., making this mill satisfactory for many materials which cannot be reduced by other mechanical means. Ground material must pass through a sieve dovetailed into frame above receiver.

Furnished with cast aluminum drawer, 28 oz. capacity, for collecting sample.

**4275-R3. Wiley Laboratory Mill, Standard Model No. 3,** motor driven, mounted on enclosed base, with drawer of cast aluminum. With ½ h.p. continuous duty motor, 1725 r.p.m.; starting switch with thermal overload cutout; V-belt, belt guard; three sieves with openings of ½ mm, 1 mm and 2 mm diameter. For 115 volts, 60 cycles, single phase, a.c. Without Stand or Tray **\$76.50**

**4275-F6. Spillage Tray Attachment,** for use with above, consisting of bracket and Stainless steel tray..... **23.00**

**4275-Z. Portable Stand,** 10 inches high, for mounting above Mills. Consisting of rubber insulated platform 23 ½ inches square, mounted on ball-bearing, swivel casters with foot-operated wheel brakes..... **119.00**

Copy of Bulletin 129 sent upon request.

**ARTHUR H. THOMAS COMPANY**

More and more laboratories rely on Thomas Laboratory Apparatus and Reagents  
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CIRCLE 1047 ON READER SERVICE CARD

## NEWS NOTES ON Laboratory Supplies and Testing Equipment

Note—This information is based on literature and statements from apparatus manufacturers and laboratory supply houses. The Society is not responsible for statements advanced in this publication.

### LABORATORY ITEMS

**Triocular Microscope**—A new line of Dynoptic microscopes equipped with triocular bodies has been announced. These new models provide considerable expansion of the standard line of research and laboratory instruments.

*Bausch & Lomb Optical Co.* 1711

**Prism Grating**—A new prism-grating infrared spectrophotometer, the Beckman IR-7, offering high resolution, variable scanning speed, repetitive scanning, horizontal strip-chart recording with variable abscissa and ordinate expansions, and convenient switching from double-beam to single-beam operation is available.

*Beckman Instruments, Inc.* 1712

**Bench Burner**—A new gas-oxygen bench burner for general purpose use in laboratories and glass shops has been developed.

*Bethlehem Apparatus Co., Inc.* 1713

**Sound Source**—A new, quick, and reliable instrument for field calibration of

sound measurement systems has been announced. This new instrument is specifically designed to be used with standard measurement microphones.

*B & K Instruments, Inc.* 1714

**Ultra-Violet Illuminator**—New No. 1953-MK.1 Ultra-Violet Microscope Illuminator provides a rich source of ultraviolet light for research and photomicroscopy. The new unit produces 100 watts in a sealed beam concentrated spot of ultraviolet with an output ranging from 2600 to 4000 Å.

*Burton Manufacturing Co.* 1715

**Runout Checker**—Device for checking the runout or eccentricity of both top and bottom spinning rolls and roving rolls in textile mills has been announced.

*Custom Scientific Instruments, Inc.* 1716

**Probe**—A liquid probe making possible direct reading of the dielectric constant and dissipation factor of most non-conductive liquids is available.

*Delsen Corp.* 1717

**Dynamometers**—Addition of a red maximum pointer as standard equipment to its line of traction dynamometers has been announced. Also the development of a new extra-heavy-duty model dynamometer featuring a capacity of 0-150,000 lb has been announced.

*W. C. Dillon and Co., Inc.* 1718

**Torque Balance**—Reactance and stall torques of small motors used in precision gyroscopes can be accurately measured with the Model 2880 Torque Balance.

*Eder Engineering Co.* 1719

**Stroboscope**—This is a scientific instrument useful for timing fly wheels and balance wheels in machinery, or for timing any constant speed, continuous cycle motion. It is useful in industry, the workshop, the laboratory—enables the user to obtain instantaneous views of objects in rapid motion.

*Edmund Scientific Co.* 1720

(Continued on page 86)

**CENCO®**

**COULOMETRIC TITRATOR**

FOR RAPID AUTOMATIC TITRATIONS



Measures both mercaptans and olefins accurately... automatically Simple to operate. Titrates wide range of concentrations Registers reaction time directly in tenths of a second.

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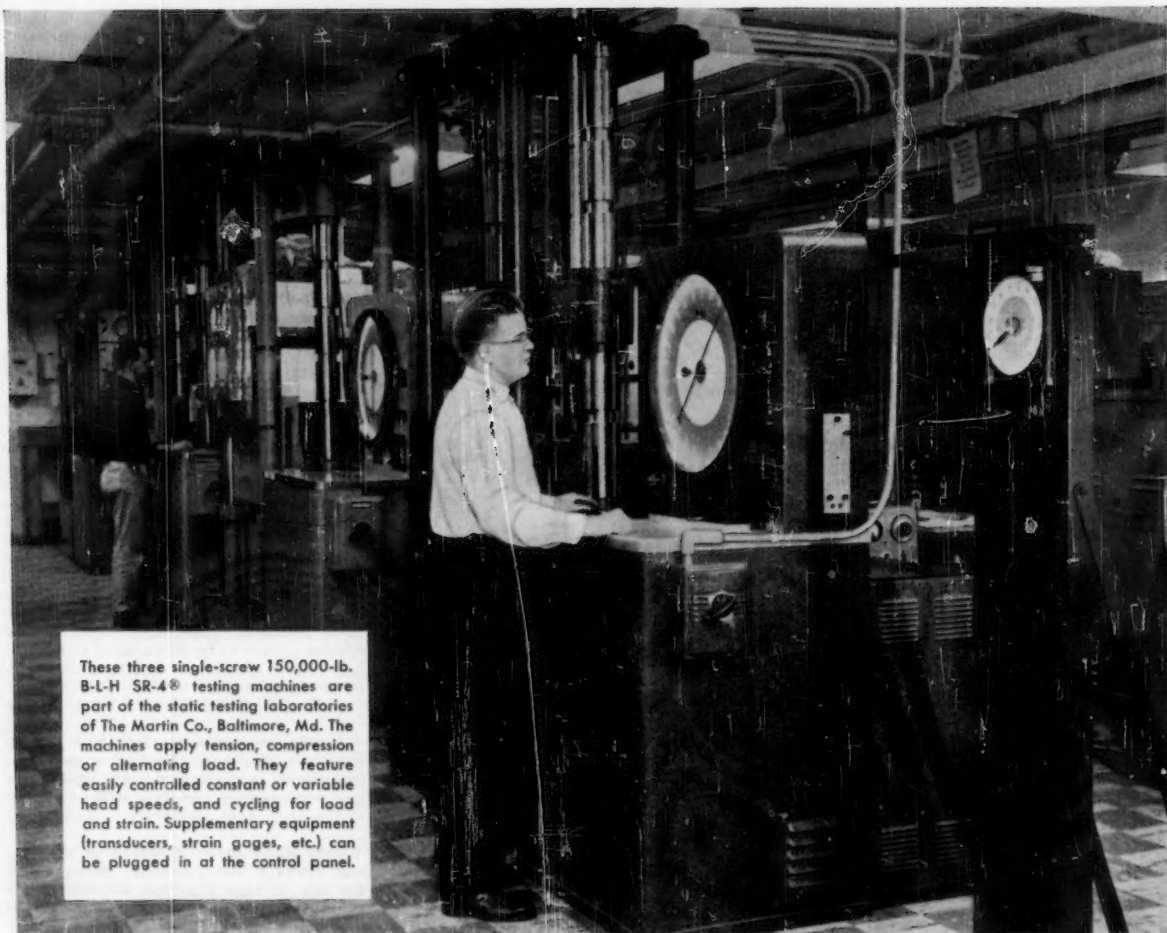
179 East 87th Street

New York 28, N. Y.

CIRCLE 1049 ON READER SERVICE CARD

CIRCLE 1049 ON READER SERVICE CARD





These three single-screw 150,000-lb. B-L-H SR-4® testing machines are part of the static testing laboratories of The Martin Co., Baltimore, Md. The machines apply tension, compression or alternating load. They feature easily controlled constant or variable head speeds, and cycling for load and strain. Supplementary equipment (transducers, strain gages, etc.) can be plugged in at the control panel.

## Martin labs use B-L-H equipment in 6-year program of fatigue, static, creep tests

As part of the preplanning on an enormous testing program to support design work on aircraft and missiles, The Martin Co., of Baltimore, Md., outlined its extensive requirements to B-L-H. Because exhaustive testing is a vital part of the aircraft industry, the equipment used must be versatile, accurate and durable. Tests are both static and dynamic and include tension, compression, flexure, creep and fatigue.

Equipped with 28 B-L-H testing machines in the static and fatigue laboratories of its Baltimore plant alone, The Martin Co. has just completed a massive 6-year testing program successfully. Its B-L-H fatigue machines have operated constantly, day and night, 7 days per

week for 6 years, requiring no maintenance except lubrication. With B-L-H creep testers in conjunction with a photographic measuring process originated at Martin, creep deformation is read to a sensitivity of .00001 in. And the SR-4 testing machines (shown above) duplicate on test components the entire strain history of an aircraft. They can also hold constant loads and they have eliminated the need for three shifts of operators working for 3 days to supervise 72-hour tests.

Whatever your testing requirement . . . compression, tension, creep, fatigue, impact or torsion . . . see B-L-H first. Or write 4-J and ask to have a B-L-H man call on you.

**BALDWIN · LIMA · HAMILTON**  
Electronics & Instrumentation Division  
Waltham, Mass.

SR-4® Strain Gages • Transducers • Testing Machines  
FOR FURTHER INFORMATION CIRCLE 1050 ON READER SERVICE CARD



## Laboratory Items

(Continued from page 84)

**Hand Spectroscope**—A compact, sturdily built instrument using diffraction grating replica of 13,400 lines per inch has been announced. It is easily carried in pocket.

*Edmund Scientific Co.*

1721

**Thermistors**—Use of thermistors has been extended to new applications by the development of beads to function continuously at 1200 F. It is believed that these are the highest temperature thermistors commercially available.

*Fenwal Electronics, Inc.*

1722

**Partitioner**—A gas chromatography instrument that makes separations at column temperatures as low as 0 C and as high as 300 C is now at service. The new Model 300 Fisher-Gulf Partitioner has 9-working temperatures.

*Fisher Scientific Co.*

1723

**Micrometer Slide Cathetometer**—Recently introduced is a new micrometer slide cathetometer specially designed for use in college physics laboratories. The new instrument, Model M940-301, enables college physics laboratories to provide students with valuable experience in the use of precision instruments.

*Gaertner Scientific Corp.*

1724

**Hazemeter**—Designed for the measurement of haze and luminous transmittance of transparent plastics and other materials. Special model is available for measuring reflectance of aluminum—meets requirements of ASTM Method D 1003 - 52.

*Gardner Laboratory, Inc.*

1725

**Colorgard**—A new automatic colorimeter for production control of the color of raw cotton has been announced. This new instrument is also adaptable to production control of the color of plastics, ceramics, etc.

*Gardner Laboratory, Inc.*

1726

**Chromanalyzer**—A compact new bench-model U-V chromatographic analyzer designed specifically for the Fluorescent Indicator Adsorption Method (ASTM Method D 1319) has been announced.

*Jarrell-Ash Co.*

1727

**Reaction Flasks**—Small reaction flasks have been added to the Bantam-ware line of small organic glass apparatus. Top and bottom flask sections are interchangeable for six different flask assemblies in the range of 50 to 500 ml capacity.

*Kontes Glass Co.*

1728

**Generator**—The new ADL Liquid Nitrogen Generator System provides a complete nitrogen liquefying facility. This system proves to be economical to laboratory users who have moderate demands for liquid nitrogen.

*Arthur D. Little, Inc.*

1729

**Nondestructive Inspection**—New, multi-frequency equipment for high-speed, non-destructive inspection of both non-ferrous and nonmagnetic metals is now available.

*Magnetic Analysis Corp.*

1730

**Test Resistance**—Addition of synchro and resolver test resistance networks to the line of high precision measuring devices is announced. The networks are designed for convenient testing of synchros and resolvers to determine electrical error by the proportional voltage method.

*Minneapolis-Honeywell Regulator Co.,  
Rubicon Instruments Division*

1731

**Well Counter**—A new well-type scintillation counter for research and medical use designed to detect all levels of gamma radiation in prepared samples has been announced.

*Nuclear Measurements Corp.*

1732

**Geiger Tube**—A large range of alpha-beta-gamma end-window geiger tubes, of varying window thicknesses, is now available. These include detectors with bismuth mesh cathodes that provide high sensitivity to gamma radiation, especially to the low energy gamma rays from iodine-131.

*Nucleonic Corporation of America*

1733

**Paper Thermometers**—Thermopaper and thermotube disposable paper thermal indicators are available for 100, 105, and 110 F to supplement the previously offered range of 115 through 490 F.

*Paper Thermometer Co.*

1734

**Wet Film Applicator**—This wet film applicator is used in conjunction with "hiding power" charts, plate glass, or any other smooth, flat surfaces and on these surfaces can be laid any sheet material, flexible or otherwise. It is a simple, yet accurate, experimental or quality control gage and there are no adjustments to make.

*Precision Gage and Tool Co.*

1735

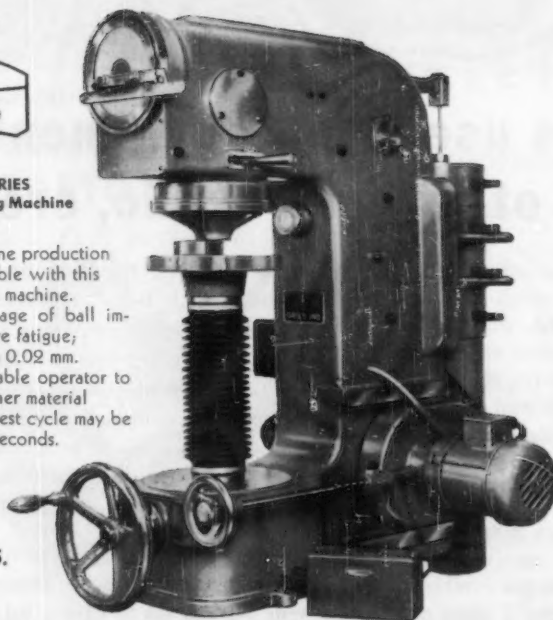
# Now, STANDARD Brinell method test results . . . in a motorized production testing machine. Shows **AUTOMATICALLY** the **DIAMETERS** of the ball impressions



### THE WOLPERT-GRIES Brinell Hardness Testing Machine Reflex Type

Accurate, rapid routine production Brinell tests are possible with this motorized Reflex type machine. Greatly magnified image of ball impression eliminates eye fatigue; increases precision to 0.02 mm. Simple limit stops enable operator to tell at a glance whether material meets specifications; test cycle may be varied from 6 to 60 seconds.

Write for  
Bulletin No. A-15.



**GRIES INDUSTRIES, INC.**  
Testing Machines Division • New Rochelle 2, N. Y.

FOR FURTHER INFORMATION CIRCLE 1051 ON READER SERVICE CARD

**Level Gage**—Continuous Level Gage, Model 40104, measures the level of liquids in totally enclosed containers of any size and dimensions with accuracy to a few hundredths of an inch. Continuous readings are provided in depth for volumes.

*Radiation Counter Laboratories, Inc.*

1736

**Sample Applicator**—A new design of sample applicator with heated airstream for quick and accurate setup of paper chromatograms by pipetting is announced.

*Research Specialties Co.*

1737

**Illuminator**—A new surface illuminator which can be used for all standard magnifications, namely 10 to 100X, on microprojector, is introduced.

*George Scherr Co.*

1738

**Emission Spec Samples**—Powder standards for the qualitative and semiquantitative determination of elements using emission spectroscopy, and a new set of standards containing the rare earth elements has been announced. Consisting of accurately measured amounts of fourteen rare earth elements plus yttrium and scandium blended into a high-purity lithium carbonate base, the standards are used for determining these sixteen elements in ores, precipitates, concentrates, alloys, etc.

*Spez Industries, Inc.*

1739

**Laboratory Balance**—The Troemner 400 offers convenience features that further simplify and speed determinations requiring sensitivity as close as 2 mg. A new, easily-read single pointer and a wider graduated beam are clearly visible for weighing with the lid closed.

*Henry Troemner, Inc.*

1740

**Grating Accessory**—The accessory for the Unicam SP. 100 Infrared Spectrometer employs two gratings of the Merton replica type, ruled at 1500 and 3000 lines per inch, for the ranges 650-2150 and 2150-3650  $\text{cm}^{-1}$ .

*Unicam Instruments, Ltd.*

1741

**Ionization Gauge**—A new flanged all-metal Bayard-Alpert type ionization gauge with nonburnout iridium filament (FG-200), or double tungsten filament (FG-200-T) is now available.

*Veeco Vacuum Corp.*

1742

**Coaxial Terminations**—New SC 50 ohm coaxial terminations has unique feature frequency range from dc to 10 kmc with SC male and female connectors.

*Weinschel Engineering*

1743

**Industrial X-Ray**—A new 150-250 kv industrial -ray control, which can produce clearly defined images through  $\frac{1}{4}$  in. of aluminum or 4 in. of steel, is available. Offering a range of 30 to 250 kv, it is provided with a 150-kv transformer or used as a 250-kv control when coupled with a 250-kv transformer.

*Westinghouse Electric Corp.*

1744

#### CATALOGS & LITERATURE

**Temperature Test Chamber**—A new environmental test chamber for low-high temperature testing in the range of -100 to +800 F is described in *Bulletin C-12*.

*Associated Testing Laboratories, Inc.*

2457

**Analytical Resins**—Data sheet on specially refined analytical grade ion-exchange

resins from Dowex materials are covered in a new *Price List "M."*

*BIO-RAD Laboratories*

2458

**Temperature Programming**—An 8-page bulletin No. 838 giving facts about temperature control in gas and vapor chromatography has been released. Illustrated by tables and curves, questions are posed and answered about column temperature, retention time, detector temperature, resolution, and temperature programming.

*Burrell Corp.*

2459

**Catalog**—One of the most complete catalogs ever produced in the laboratory supply field has been published. The completely revised and specially engineered 1273-page purchasing tool will provide buyers of laboratory supplies and equipment with more effective product descriptions, wide selection of laboratory items, faster finding aids and more efficient and convenient source for handling requisitions.

*The Chemical Rubber Co.*

2460

(Continued on page 88)

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FISHER CATALOG

59



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B-868

FOR FURTHER INFORMATION CIRCLE 1052 ON READER SERVICE CARD



## Catalog & Literature

(Continued from page 87)

**Mass Spectrometers**—Two bulletins, *Bulletin 1843A* and *Bulletin 1824C* describe Mass Spectrometers.

*Consolidated Electrodynamics Corp.* 2461

**Corrosion Measurement**—A technical paper *TR 502* describing a new technique for measuring corrosion in processing equipment is now available.

*Crest Instrument Co.* 2462

**Cement Testing**—*Bulletin 25* describes cement and mortar testing apparatus.

*Humboldt Manufacturing Co.* 2463

**Universal Tester**—An illustrated, two-color six-page folder, describing the multi-range universal tester has been released.

*W. C. Dillon and Co., Inc.* 2464

**Single-Pan Balances**—For the first time, all of the single-pan balances in the Mettler line of Gram-atic balances are described and illustrated in a single bulletin *FS-207*.

*Fisher Scientific Co.* 2465

**Microscope Accessories**—Twelve-page catalog describes microscope accessories for the entire field.

*Ernest F. Fullam, Inc.* 2466

**Acid Resistant Ware**—New 20-page *Catalog No. G 358* covering polyethylene and nylon products, not affected by concentrated acids, oxidizing agents, hydroxides, and polar liquids is available. It illustrates and describes 95 products.

*General Scientific Equipment Co.* 2467

**Strain Gauge**—*Bulletin PSG-1* "Glenite Strain Gauge Pressure Transducers," describes the performance, application, mechanical and electrical specifications, compatibility in installations, and instructions for ordering the instruments. Also included are circuit diagrams, a typical installation, and a basic explanation of the operation of the equipment.

*Gulton Industries, Inc.* 2468

**Temperature Detectors**—Complete information about Thermohm detectors (resistance thermometers) for temperatures up to 1000 F is now available in new 36-page *Catalog EN-S4*, just published.

*Leeds & Northrup Co.* 2469

**Potentiometers**—A new six-page *Data Sheet E-33(4)* describing both thermocouples and standardizing potentiometers is now available.

*Leeds & Northrup Co.* 2470

**Transistor Servo Amplifier**—Described in a two-page brochure, Librascope's miniaturized transistor servo amplifier features  $\frac{3}{4}$ -w output over a wide range of temperatures.

*Librascope, Inc.* 2471

**Instrumentation Recorder**—A new technical 4-page brochure describing the characteristics, specifications, and operating features of the new MINCOM Model C-100 series of instrumentation tape recorders is available.

*Minnesota Mining and Manufacturing Co., MINCOM Division* 2472

**Microwave Catalog**—A new 84-page catalog describing the expanded line of

microwave and UHF test equipment and components has been published.

*Narda Microwave Corp.* 2473

**Ratemeter**—New two-page bulletin describes instrument used for measuring radioactivity sensed by various types of detectors.

*Nuclear Measurements Corp.* 2474

**Microradiography**—A new 16-page booklet, *Volume 19*, giving technical facts on contact microradiography for research in biological, paper, textile, metallurgical and foodstuff fields, is available.

*Philips Electronics, Inc.* 2475

**Chromatography**—A new 4-page, 4-color bulletin on "A New Dimension in Paper Chromatography" has been announced.

*Precision Scientific Co.* 2476

**Ovens**—A new descriptive bulletin on Precision-Freas "Hi-Temp" Mechanical Convection Ovens is released. This product provides laboratory oven control with heat to 1000 F in 105 minutes.

*Precision Scientific Co.* 2477

**Radiochemical Catalog**—Production of 38 new carbon-14 labeled compounds is announced in the 1958 edition of the Radiochemical Price List of carbon-14 compounds.

*Research Specialties Co.* 2478

**Soil Testing**—Thirty-two new catalog items for soils, concrete, and asphalt are described in a new products bulletin now available. Included in this 4-page bulletin

**THE NEW VERSATILE  
NON-DESTRUCTIVE  
COATING-THICKNESS  
TESTER**

## DERMITRON



Unit Process Assemblies, Inc., pioneers in non-destructive testing and specialists in electronics for metal finishing, offer their latest DERMITRON D-2 with these features:

- Measures plated coatings on steel, brass, copper, zinc die-cast, aluminum, bronze and other metals; also, nickel on steel. ■ Measures anodize and hard-coat on aluminum and magnesium; also, paint, porcelain, organic coatings on non-ferrous metals. ■ Measures metal coatings on plastics, ceramics and other non-metallic materials. ■ Available with FOUR measuring probes for extra-wide thickness ranges from thin to thick deposits. ■ Only  $\frac{1}{8}$ " circle area required for measurement. ■ You get fast, accurate, direct readings, plus versatility and portability. ■ Sorts metals and alloys.

Write for latest brochure and questionnaire to help solve your thickness testing problems.

**UPA** **UNIT PROCESS ASSEMBLIES, INC.**  
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CIRCLE 1053 ON READER SERVICE CARD

## NEW! CENCO® FORCED CIRCULATION RECTANGULAR OVENS



All stainless steel interiors. Temperature range 60-250°C. Fitted to take glass inner door. Model 95375, 12"x12"x14" inside . . . \$315.00. Model 95380, 17"x15"x20" inside . . . \$395.00.

Write for Bulletin 5B.

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CIRCLE 1054 ON READER SERVICE CARD

tin are descriptions of the Right-A-Weigh balance, the basic concrete tester, a new 200-ton pile loading ram, a concrete air indicator, and precision air entrainment meters.

*Soiltest, Inc.*

2479

**Computer**—A new brochure is available on digital computer control and data logging. Included in the reference bulletin is a description of the RW-300 Digital Control Computer as well as comprehensive discussions of process control, data logging, pilot plant, and test facility applications for computer control systems.

*Thompson-Ramo-Wooldridge Products Co.*

2480

**Book Catalog**—Books on science, technology, and business described in 150-page catalog is available.

*D. Van Nostrand Co., Inc.*

2481

**Environmental Chambers**—New 28-page catalog gives comprehensive data including tabular information. Chambers and accessories are fully described.

*Webber Manufacturing Co., Inc.*

2482

**Loss Test Set**—An 8-page catalog, *Application Notes No. 4*, describing the Dual Channel Insertion Loss Test Set has been released.

*Weinschel Engineering*

2483

#### NEWS OF LABORATORIES

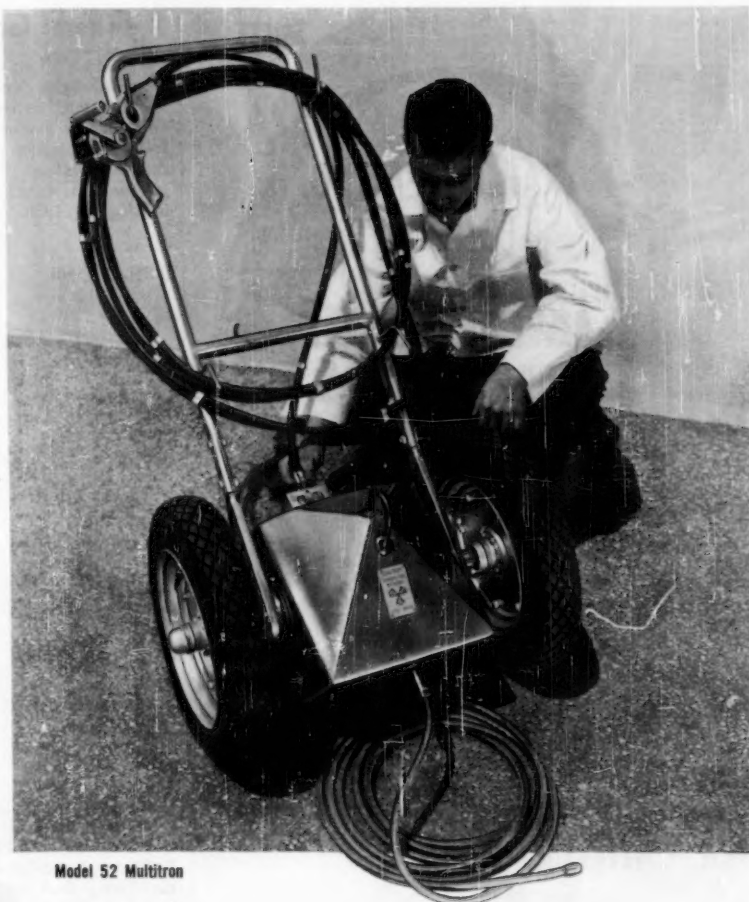
**Truesdail Laboratories, Inc., Los Angeles, Calif.**—Has opened a new laboratory at 712 Tuna Street on Terminal Island, California. The new laboratory will be equipped with the facilities formerly used at Honolulu. Left in Honolulu will be a branch office.

#### INSTRUMENT COMPANY NEWS

**Bruel & Kjaer, Naerum, Denmark**—Bruel & Kjaer of Naerum, Denmark, internationally recognized manufacturer of high quality sound, vibration, and instrument systems, has announced the appointment of a new Marketing Agency for the United States. A new corporation, B & K Instruments, Inc., is now the exclusive Marketing Agency for Bruel & Kjaer. It is headed by James W. Day, who has been active in this field of instrumentation for the past five years.

**Schaar and Co., Chicago, Ill.**—Chemical Laboratory Supply firm at 7300 W. Montrose, announces the election of L. A. Rauch as president. Rauch succeeds C. E. Schaar who retires after 48 years of service and who has long been prominent in the field of industrial and educational laboratory supply. Rauch, who holds a degree in chemical engineering from the University of Illinois, has been associated with Schaar and Company for over 24 years, most recently serving in the capacity of vice-president in charge of general sales.

**Tatnall Measuring and Nuclear Systems, Ltd., Canada**—Tatnall Measuring and Nuclear Systems Ltd. is the new name for a company formerly known as Non-Destructive Testing Corporation. The company is Canadian and a distributor for Tatnall and Nuclear Systems Products (subsidiaries of Budd Corp.) as well as Sperry Products, Inc., and J. W. Dice equipment.



Model 52 Multitron

### "We Save More Than 50% of Radiography Costs on Welding Inspection by Using Nuclear Systems' Equipment"

This statement was made recently by Mr. J. C. Abbott, General Superintendent of Manufacturing at the Chattanooga, Tennessee plant of Combustion Engineering Inc., manufacturers of steam generating and related equipment, nuclear power systems and other products.

"In inspecting welding, we're using two Nuclear Systems gamma radiography machines 18 hours a day," continued Mr. Abbott. "Soon we'll have them in operation 24 hours a day at which time we expect to

save up to 75% on radiography costs when compared with X-ray.

"In using X-ray we required expensive rigging to bring the work to the machine; now we simply move the easily portable Nuclear Systems machines around to our work locations."

Call on Nuclear Systems for your radiography equipment needs. Offices in Philadelphia, Chicago, San Francisco and Los Angeles. Sales representatives in principal cities. Catalog on request.

Visit our booth at the National Metal Exposition, Cleveland, Oct. 27-31



FOR FURTHER INFORMATION CIRCLE 1055 ON READER SERVICE CARD



## Shore DUROMETER

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- 

The Shore Durometer is available in various models for testing the entire range of rubber hardness.

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Made by the manufacturers of the "Scleroscope", for testing the hardness of metals.

**Shore** INSTRUMENT & MFG. CO., INC.  
90-35A VAN WYCK EXPRESSWAY, JAMAICA 35, N. Y.

CIRCLE 1057 ON READER SERVICE CARD

## INITIATIONS

Title	Type of Action	Symbol or Number	FSC Code	FSSC Class	Assigned Agency & Preparing Activity
Aluminum Alloy Ingot (for Remelting)	Rev.	QQ-A-371c	..	..	DOD-Army-USAF
Amyl Alcohol; Secondary (for use in Organic Coating)	Rev.	TT-A-516b	6810	..	GSA-FSS
Carpets and Rugs, Wool-Nylon, Loop Pile, Woven, Attached Rubber Cushioning	New	DDD-C-85a	7220	72	GSA-FSS
Ethyl Alcohol	Int.	O-E-760b	6505	..	GSA-FSS
	Am. 1		6810		
Insulating Oil, Electrical (for Transformers, Switches and Circuit Breakers)	New	VV-T-530	9160	..	DOD-Navy-Aer
Isopropyl Acetate (for use in Organic Coatings)	Rev.	TT-I-721b & TT-I-00721a (GSA-FSS)	6810	..	GSA-FSS
	New				
Lubricating Oil, Refrigerant	Am. 1	VV-L-825	9150	..	DOD-Navy-Aer
Plating, Cadmium (Electrodeposited)	Am. 1	QQ-P-416a	..	..	DOD-Navy-Ord.
Thread, Cotton	Am. 2	V-T-276d	8310	83	GSA-FSS
Thread, Linen	Am. 1	V-T-291b	8310	83	DOD-Army-QMC
Thread, Silk	Am. 1	V-T-301a	8310	83	DOD-Army-QMC

[illegible]

Write  
for Our  
New  
138-Page  
Catalog!

**DELTA CHEMICAL WORKS, Inc.**  
23 West 60th St. New York 23, N. Y. PLaza 7-6317

CIRCLE 1058 ON READER SERVICE CARD



## PROMULGATIONS

Title	Type of Action	Symbol or Number
Anodes, Copper (superseding QQ-C-493)	New	QQ-A-673
Asphalt; Cut-Back (for) Road-Work	Am. 1	SS-A-671a
Cloth, Cotton, Broadcloth, Mercerized (superseding CCC-C-00437 (GSA-FSS) & CCC-B-686a)	New	CCC-C-437a
Drums, Fiber, with Recessed Ends		
Drums, Fiber (superseding PPP-D-00723b (COM-BDSA))	New Rev.	PPP-D-715 PPP-D-723c
Drums, Metal, Reconditioned, 55-Gallon (for Shipment of Noncorrosive Material) (superseding PPP-D-00732 (COM-BDSA))	New	PPP-D-732a
Sieves, Standard for Testing Purposes	Am. 1	RR-S-366b
Thread, Cotton	Am. 1	V-T-276d
Thread, Cotton Gimp, Buttonhole	New	V-T-280
Tubes, Copper, Seamless, 6000 psi Maximum Pressure (superseding WW-T-797)	Rev.	WW-T-797a
Water-Repellent Compound, Textile Finish	New	TT-W-156
Zinc; Sheet and Strip	Am. 1	QQ-A-301b

Paint, Poly(Vinyl Acetate) Emulsion, Exterior  
Paint, Styrene-Butadiene Emulsion, Exterior  
Sodium Carbonate-Sodium Bicarbonate Mixture

New  
New  
New

TT-P-0055a (Army-CE)  
TT-P-0099a (Army-CE)  
P-S-00641e (GSA-FSS)

## CANCELLATIONS

Title	Symbol or Number	Reason for Cancellation
Broadcloth; Cotton, Mercerized	CCC-B-686a	Superseded by Fed. Spec. CCC-C-437a
Bronze, Aluminum; Bars, Plates, Rods, Shapes, Sheets, and Strips	QQ-B-666	Superseded by Fed. Spec. QQ-B-663 & Fed. Spec. QQ-B-667
Copper; Anodes	QQ-C-493	Superseded by Fed. Spec. QQ-A-673
Oil; Linseed, Heat-Polymerized (Bodied), for Paint, Varnish, and Enamel	TT-O-367	Superseded by Fed. Spec. TT-L-201
Osanburg; Cotton	CCC-O-721	Superseded by Fed. Spec. CCC-C-429

## INTERIM FEDERAL SPECIFICATIONS AND STANDARDS ISSUED

Title	Type of Action	Symbol or Number
Amyl Alcohol; Secondary (for Use in Organic Coatings)	New	TT-A-00516(GSA-FSS)
Boxes, Fiber	New	PPP-B-00636a (Army-QMC)
Carpet and Rug, Wool Loop Pile, Knitted	New	DDD-C-0080(GSA-FSS)
Carpets and Rugs, Wool-Nylon, Loop Pile, Woven, Attached Rubber Cushioning	New	DDD-C-0085(GSA-FSS)
Isopropyl Acetate (for Use in Organic Coatings)	New	TT-I-00721a(GSA-FSS)
Paint, Acrylic Emulsion, Exterior	New	TT-P-0019a (Army-CE)
Paint, Odorless, Alkyd, Interior Flat White and Tints	New	TT-P-0030a (Army-CE)

## SPECIFICATIONS AND STANDARDS APPROVED FOR PRINTING

Title of Specification	Type of Action	Symbol or Number
Acetic Acid, Glacial, Technical	Rev.	O-A-76c
Aluminum Bronze Bars, Rods, Shapes, Drawn Strip, and Forgings	New	QQ-A-630
Bags; Textile, Shipping, Burlap, Cotton, and Waterproof Laminated	Am. 1	PPP-B-35
Bronze, Aluminum; Rods, Bars, Shapes, and Forgings	Canc.	QQ-B-663
Methanol, Technical (Methyl Alcohol)	Rev.	O-M-232b
Wire, Electrical; Steel, Copper-Covered	New	QQ-W-345
Wire, Steel, Copper-Covered	Canc.	QQ-W-421a

## CUSTOM WORK SERVICE ENGINEERING INSTRUMENTS & EQUIPMENT

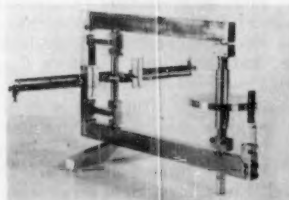
The console is equipped with three cells developed by Plastics Technical Service of the Dow Chemical Co. The cell measures the gas transmission rate of plastic sheeting and plastic coated paper. The gas transmission rate is determined from measurements of pressure and volume change of gas transmitted through the specimen.

Ref: ASTM Designation D-1434-56T

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541 DEVON STREET  
KEARNY, N. J.



Dow Gas Transmission Cell Console



Templin Calibrator



Accelerated Soil Tester



High Speed Angle Centrifuge



CSI Stoll QM Univ. Wear Tester

FOR FURTHER INFORMATION CIRCLE 1059 ON READER SERVICE CARD

## The Bookshelf

(Continued from page 82)

### Heating Ventilating Air Conditioning Guide 1958

American Society of Heating and Air-Conditioning Engineers, 62 Worth Street, New York 13, N. Y.; 36th Edition (1958); 1775 pp.; \$12.

THE 1958 guide, containing a 1272 page Technical Data Section and a 503 page Catalog Data

Section, is the largest annual edition published. In planning this edition it became necessary to again expand the Technical Data Section to include the worth-while and needed technical data made available since the previous edition.

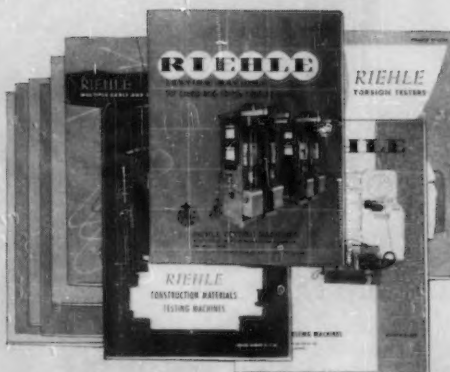
These revisions and additions include (1) addition of both a description and a design method for high velocity air duct systems, (2) an enlarged section on the heat pump, (3) new information on exhaust hood performance and design for hot and cold processes, (4) a

simplified presentation of industrial drying principles, calculations, and system design, (5) extension of data on heavy fuel oils and their use, with pre-heating, in automatic fuel burning equipment, (6) new data on heat gain through glass block used in skylights, requirements for shading glass, and basic principles involved in calculating heat flow through glass area, (7) a general revision of the chapter on radiators, convectors, baseboard, and finned-tube units and addition of new data on their ratings, and performance, (8) a revision of ranges of capacity for electrical heating units and a method of determining operating cost for these units, (9) an enlarged section on performance and testing of air cleaners, (10) extension of list of allowable concentrations of air contaminants, (11) an increased number of codes and standards of interest to users of The Guide.

The Catalog Data Section follows the pattern of other editions, but the equipment data have been brought up to date.

## now complete your Riehle testing machine reference file...

You can take the first step toward solving your materials problems by bringing your Riehle testing machines catalog file up to date. Check the list below, clip it to your letterhead, and we'll send the catalogs you request promptly, without obligation.



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| <input type="checkbox"/> Universal Screw Power Testing Machines and Accessories  | <input type="checkbox"/> Horizontal Tensile Testing Machines    |
| <input type="checkbox"/> Construction Materials Testing Machines and Accessories | <input type="checkbox"/> Impact Testing Machines                |
| <input type="checkbox"/> Torsion Testing Machines                                | <input type="checkbox"/> Testing Machines Guide                 |
| <input type="checkbox"/> Creep and Stress-Rupture Testing Machines               | <input type="checkbox"/> Riehle Recorder and Accessories        |
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| <input type="checkbox"/> Brinell Hardness Testers                                |   |
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| <input type="checkbox"/> Portable Hardness Testers                               |   |

**Riehle TESTING MACHINES**  
A DIVISION OF  
**American Machine and Metals, Inc.**  
EAST MOLINE, ILLINOIS

### National Fire Codes

National Fire Protection Assn., 60 Battery-march St., Boston 10, Mass. 1958; Six Volumes

THE 1958 edition of the National Fire Codes is now available. This consists of six volumes covering:

- (Vol. I) Flammable liquids and gases;
- (Vol. II) Combustible solids, dusts, etc.;
- (Vol. III) Building construction and equipment;
- (Vol. IV) Extinguishing equipment;
- (Vol. V) Electrical; and
- (Vol. VI) Transportation, etc.

The codes are compilations of standards developed by the N.F.P.A. and are purely advisory but are widely used as a basis of good practice.

Volume III on Building Construction and Equipment is of particular interest to ASTM members. The many facets of building construction and equipment in which fire hazards are present are covered in the various chapters, including such as protection of openings in walls and partitions; incinerators; heating and air conditioning systems; garages; waterproofing; chimneys, flues, and vents; and construction operations.

The several ASTM standards on fire tests of building construction and materials are presented in full in this volume.

The volumes are published by the National Fire Protection Association, a non-profit technical and educational association devoted to the reduction of loss of life and property by fire; further information may be obtained from the N.F.P.A.

FOR FURTHER INFORMATION CIRCLE 1060 ON READER SERVICE CARD

## New Foreign Technical Information Center Announced by Department of Commerce

A FOREIGN Technical Information Center is now operating in the U. S. Department of Commerce to provide American science and industry with access to translations of a large amount of Soviet technical information, according to a recent announcement by Secretary of Commerce Sinclair Weeks.

The center is a part of the Office of Technical Services, Business and Defense Services Administration, with John C. Green as director of OTS.

The services of the Foreign Technical Information Center include publication of abstracts of all articles appearing in 141 Soviet technical journals, translations of important sections of *Referativny Zhurnal* (the Russians' own abstract journal), and a semi-monthly review of various areas of Soviet science compiled by the Central Intelligence Agency. Abstracts of each issue of the 141 journals may be purchased from OTS on a subscription or single issue basis, as may CIA's *Scientific Information Report* (see OTS Reports below). The various sections of *Referativny Zhurnal* will be sold initially by single issues, but subscription sales may be offered later.

OTS will soon begin distributing complete translations of articles and books, according to Mr. Green.

Congress appropriated \$510,000 for the operation of the foreign technical information program in fiscal 1959. Functions of the program are to collect, catalog, print, and sell abstracts and complete translations of foreign technical documents.

A mimeographed list of publications of the Foreign Technical Information Center, including prices, is available from the U. S. Department of Commerce, Office of Technical Services, Washington 25, D. C.

### OTS Research Reports

These reports, recently made available to the public, can be obtained from the Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C. Order by number.

### Publications of the Foreign Technical Information Center

OTS publishes English abstracts of Soviet technical periodicals (see story above), including the following:

### General

Scientific Information Report, PB 131891T. Issued semimonthly, \$28 a year, \$2.75 single issue. A general review of current Soviet scientific developments compiled by the Central Intelligence Agency.

### Abstract Journals

*Referativny Zhurnal* (Russian Abstract journal). Translated sections of the biology, chemistry, and physics series are available. Write to OTS for monthly price list.

### Abstracts of Soviet Technical Periodicals

All articles in each issue of the following publications are abstracted in English on abstract cards. Abstract cards may be purchased on a subscription basis at the prices indicated. Abstracts of a single issue of any publication are 50 cents per set.

### ASTRONOMY

*Progress of Mathematical Sciences*, PB 141036T. Bimonthly, \$3 a year.  
*Journal of Abstracts, Mathematics*, PB 141038T. Monthly, \$6 a year.

### CHEMISTRY AND CHEMICAL ENGINEERING

*News of the Academy of Sciences of the USSR, Department of Chemical Sciences*, PB 141055T. Monthly, \$6 a year.  
*News of Institutes of Higher Learning of the Ministry of Higher Education*, PB 141056T.  
*USSR, Chemistry and Chemical Technology*, Bimonthly, \$3 a year.  
*Raw and Vulcanized Rubber*, PB 141057T. Monthly, \$6 a year.  
*Chemical Science and Industry*, PB 141058T. Bimonthly, \$3 a year.  
*Chemical Industry*, PB 141059T. Eight issues a year, \$4 a year.  
*Chemistry and Technology of Fuels and Oils*, PB 141060T. Monthly, \$6 a year.  
*Coke and Chemistry*, PB 141063T. Monthly, \$6 a year.

(Continued on page 94)

# ACCURATE LABORATORY OR JOB-SITE TESTING...

improves quality control  
prevents over-design  
lowers project costs

ON ALL CONSTRUCTION!

The advertisement features several illustrations of testing equipment. On the left, a large vertical machine is labeled 'CH-980 VOLUMEASURE'. Above it, a smaller machine is labeled 'CL-392 SIEVE SHAKER'. Below the sieve shaker, a machine is labeled 'U-160 COMPRESSION TESTER'. In the center, there are three machines: 'CT-711 CONCRETE TESTER', 'CT-375 BEAM TESTING MACHINE', and 'CT-900 CONCRETE BLOCK TESTER'. To the right, a large machine is labeled 'AP-350 VERSA-TESTER'. A woman is shown operating this machine. Below the machines, there are labels for 'soils', 'concrete', and 'asphalt'. The text 'Engineering Test Apparatus for Soils, Construction Materials, Concrete and Asphalt ranging from single items to self-contained Mobile Laboratories are available for immediate shipment.' is present. The company name 'SOILTEST Incorporated' is prominently displayed in a stylized font. Below it, the address '4711 WEST NORTH AVENUE • CHICAGO 39, ILLINOIS, U.S.A.' is given. To the right, the 'EASTERN OFFICE' address '60 EAST 42nd ST., NEW YORK 17, N. Y.' and telephone number 'TELEPHONE YUkon 6-7383' are listed.



## OTS Reports

(Continued from page 93)

*Crystallography*, PB 141065T. Bimonthly, \$3 a year.  
*Alcohol Industry*, PB 141067T. Monthly, \$6 a year.  
*Glass and Ceramics*, PB 141068T. Monthly, \$6 a year.  
*Ukrainian Chemistry Journal*, PB 141069T. Bimonthly, \$3 a year.  
*Progress of Chemistry*, PB 141070T. Monthly, \$6 a year.  
*Journal of Analytical Chemistry*, PB 141071T. Bimonthly, \$3 a year.  
*Journal of Physical Chemistry*, PB 141072T. Monthly, \$6 a year.  
*Journal of Inorganic Chemistry*, PB 141074T. Monthly, \$6 a year.  
*Journal of General Chemistry*, PB 141075T. Monthly, \$6 a year.  
*Journal of Applied Chemistry*, Monthly, \$6 a year.  
*Journal of Abstracts, Chemistry*, PB 141077T. Semimonthly, \$12 a year.

### CIVIL ENGINEERING

*Concrete and Reinforced Concrete*, PB 141090T. Monthly, \$6 a year.  
*Hydraulic Engineering Construction*, PB 141091T. Monthly, \$6 a year.  
*Hydraulic Engineering and Soil Improvement*, PB 141092T. Monthly, \$6 a year.  
*Cement*, PB 141094T. Bimonthly, \$3 a year.

### ELECTRICAL ENGINEERING

*Electricity*, PB 141098T. Monthly, \$6 a year.  
*Radio Engineering and Electronics*, PB 141106T. Monthly, \$6 a year.  
*Herald of the Electrical Industry*, PB 141107T. Monthly, \$6 a year.  
*Herald of Communications*, PB 141108T. Monthly, \$6 a year.  
*Journal of Abstracts, Electrical Engineering*, PB 141109T. Monthly, \$6 a year.

### FUEL AND POWER

*Atomic Energy*, PB 141082T. Monthly, \$6 a year.  
*Petroleum Economy*, PB 141086T. Monthly, \$6 a year.

### MECHANICAL ENGINEERING

*Measurement Techniques*, PB 141112T. Bimonthly, \$3 a year.  
*News of Institutes of Higher Learning of the Ministry of Higher Education, USSR, Instrument Manufacture*, PB 141115T. Bimonthly, \$3 a year.  
*Scientific Reports of Higher Schools, Machine Construction and Instrument Manufacture*, PB 141118T. Quarterly, \$2 a year.  
*Instrument Manufacture*, PB 141119T. Monthly, \$6 a year.  
*Instruments and Experimental Techniques*, PB 141120T. Bimonthly, \$3 a year.  
*Journal of Abstracts, Mechanics*, PB 141124T. Monthly, \$6 a year.

### MINING AND METALLURGY

*Physics of Metals and Metallography*, PB 141126T. Bimonthly, \$3 a year.  
*Ministry of Higher Education, Ferrous Metallurgy*, PB 141128T. Bimonthly, \$3 a year.  
*Ministry of Higher Education, Nonferrous Metallurgy*, PB 141129T. Bimonthly, \$3 a year.  
*Foundry Practice*, PB 141130T. Monthly, \$6 a year.  
*Metallography and Metal Working*, PB 141131T. Monthly, \$6 a year.  
*Metallurgist*, PB 141132T. Monthly, \$6 a year.  
*Refractories*, PB 141134T. Monthly, \$6 a year.  
*Steel*, PB 141136T. Monthly, \$6 a year.  
*Steel-Casting Practice*, PB 141137T. Monthly, \$6 a year.  
*Welding Practice*, PB 141138T. Monthly, \$6 a year.  
*Nonferrous Metals*, PB 141139T. Monthly, \$6 a year.  
*Journal of Abstracts, Metallurgy*, PB 141140T. Monthly, \$6 a year.

### PHYSICS

*Journal of Acoustics*, PB 141039T. Quarterly, \$2 a year.  
*Theory of Probability and Its Applications*, PB 141048T. Quarterly, \$2 a year.  
*Progress of Physical Sciences*, PB 141049T. Monthly, \$6 a year.  
*Journal of Experimental and Theoretical Physics*, PB 141052T. Monthly, \$6 a year.  
*Journal of Technical Physics*, PB 141053T. Monthly, \$6 a year.  
*Journal of Abstracts, Physics*, PB 141054T. Monthly, \$6 a year.  
**SCIENCE AND TECHNOLOGY—GENERAL**  
*Standardization*, PB 141023T. Bimonthly, \$3 a year.

### Catalogs of Technical Reports

The CTE's list all research reports available from the OTS collection in the fields of molded plastics, polystyrene, zirconium, anodic coatings, and greases and lubricants.  
*Molded Plastics*, 1938-58. CTR-347, 10 cents.  
*Polystyrene*, 1930-58. CTR-346, 10 cents.  
*Zirconium*, 1932-58. CTR-344, 10 cents.  
*Anodic Coatings*, 1934-58. CTR-348, 10 cents.  
*Greases and Lubricants*. CTR-345, 10 cents.

### Technical Reports

*Study of the Factors Influencing the Properties of Heat Treatable, Titanium Sheet Alloys*, PB 131769. \$4.  
*Spectrographic Determination of Oxygen in Titanium*, PB 121641. 75 cents.  
*Investigation of Nodular Cast Iron Manufactured in the Springfield Armory Casting Laboratory*, PB 131399. \$1.25.  
*Alloying and Heat Treating Ductile Cast Iron, Final Report*, PB 131350. \$2.50.



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of bulk materials  
with

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## TEST SIEVE SHAKERS

The new, improved SYNTRON Test Sieve Shakers provide a more positive control of components and more uniform sieve analysis of bulk materials.

SYNTRON Test Sieve Shakers feature a larger, more powerful electromagnetic drive for rapid, positive gradation plus lower maintenance, with a rheostat and voltmeter for comparable tests under identical conditions and a reset timer for accurately timed test periods.

SYNTRON Test Sieve Shakers use 6 standard 8" test sieves and bottom pan in a coarse and fine series.

Operation is from 115 volt, 60 cycle a-c.

TS 358

Write for complete information on SYNTRON Test Sieve Shakers — FREE

**SYNTRON COMPANY**

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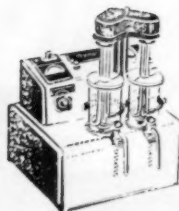
Homer City, Penna.

FOR FURTHER INFORMATION CIRCLE 1062 ON READER SERVICE CARD

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## rapid separation of metals by mercury cathode DYNA-CATH



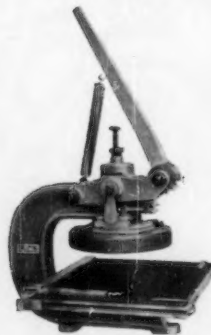
For determination of aluminum in steel, aluminum in zinc base alloys, and other separations. This apparatus does a faster, more complete job in analytical separation of metals. Example, Dyna-Cath quantitatively removes 1 gram of iron from 50 ml. of electrolyte in 20 minutes or less. Request Bulletin 220-F.

**Eberbach**  
CORPORATION

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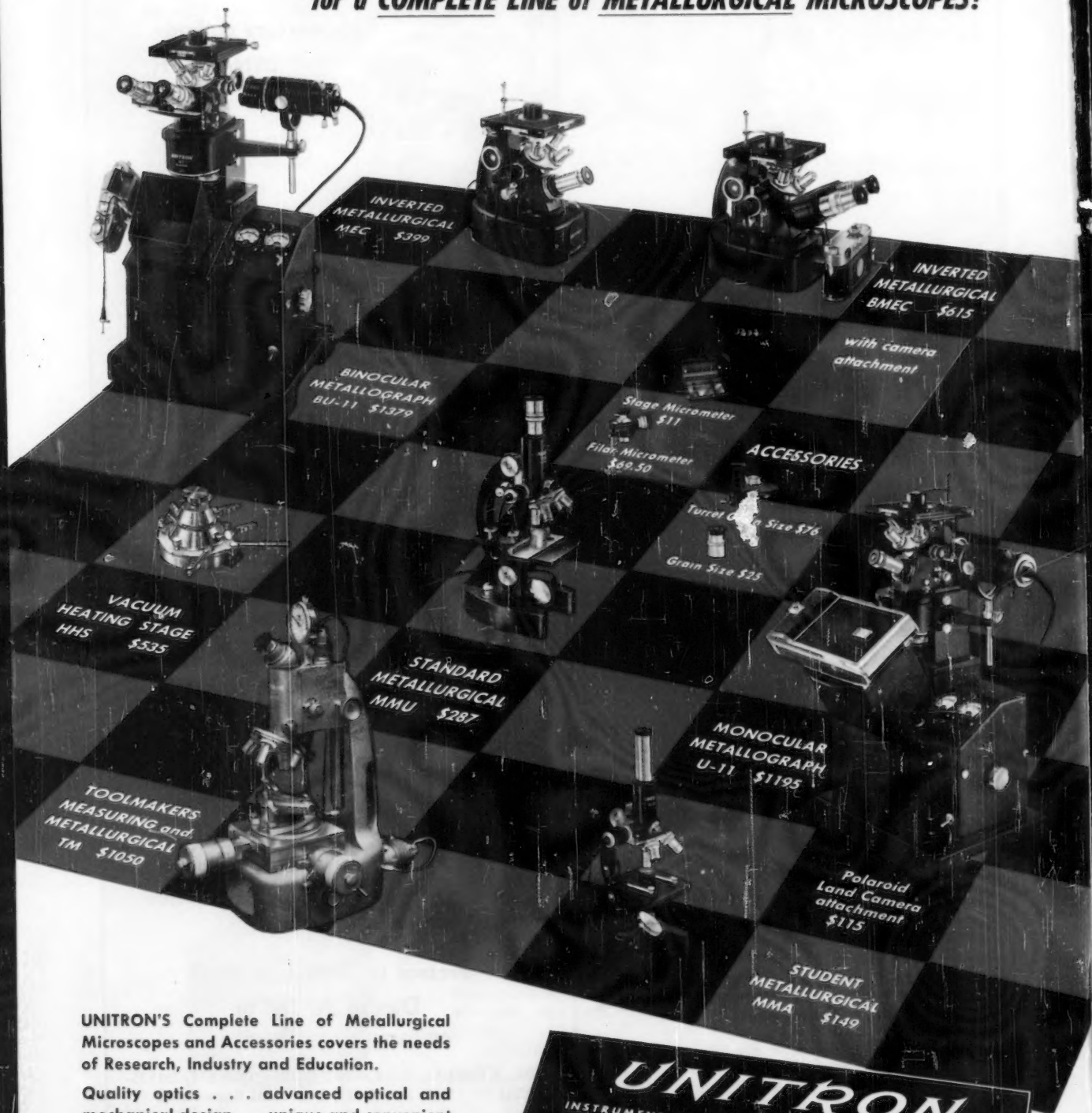
**SMS INSTRUMENT COMPANY**

P. O. Box 585, Summit, N. J.  
P. O. Box 24, Rensselaer, N. Y.

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CIRCLE 1065 ON READER SERVICE CARD

## UNITRON

INSTRUMENT DIVISION OF UNITED SCIENTIFIC CO.  
204-206 MILK STREET - BOSTON 9, MASSACHUSETTS

Please rush UNITRON's Microscope Catalog 3G.

Name

Company

Address

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Type 1603-A Z-Y Bridge...\$370

#### SPECIFICATIONS:

##### Impedance and Admittance Range

R:  $\pm 1000$  ohms G:  $\pm 1000$   $\mu$ hos  
X:  $\pm 1000$  ohms B:  $\pm 1000$   $\mu$ hos

##### Accuracy

R or G:  $\pm [1\% + (2 \text{ ohm or } 2 \mu\text{mho})]$

X or B:  $\pm [1\% + (\frac{2f_0}{f} \text{ ohms or } \frac{2f}{f_0} \mu\text{mho})]$

f is operating frequency,  $f_0$  is frequency setting of panel selector switch.

Impedances of less than  $100\Omega$  (or  $100 \mu$ hos) can be measured on "Initial Balance" dials with considerably greater accuracy —

R or G:  $\pm [1\% + (0.2 \text{ ohm or } 0.2 \mu\text{mho})]$

X or B:  $\pm [1\% + (\frac{0.2f_0}{f} \text{ ohm or } 0.2 \frac{f}{f_0} \mu\text{mho})]$

Frequency Range — 20 cycles to 20 kc

##### Maximum Applied Voltage

130 volts, rms on bridge gives less than 32 v on unknown

##### Accessories Recommended:

Type 1210-C Unit R-C Oscillator.....\$180

Type 1212-A Unit Null Detector.....\$145

Z-Y Bridge measures impedance characteristics of a "Dynapoise Drive" self-balancing recorder element. A G-R Type 1210-C Unit Oscillator is used as bridge generator, and a G-R Type 1212-A Unit Null Detector indicates bridge balance.

Photo Courtesy: The Foxboro Company

# One BRIDGE TO MEASURE Any IMPEDANCE

Ever try to identify an impedance, only to find its value outside the bridge's range? This *can't* happen if you use the General Radio Type 1603-A Z-Y Bridge — it measures *any* impedance, from short circuit to open circuit, real or imaginary, positive or negative, over the complete audio-frequency range.

Proof of the Z-Y Bridge's versatility is demonstrated by its application at The Foxboro Company, manufacturers of "Dynalog" self-balancing recorders, indicators, and controllers. These instruments are used in industrial processing for the measurement of: temperature, pressure, humidity, voltage, flow, weight, thrust, drag, displacement, and many other variables. Many of these applications require "special" Dynalog instruments with modified input characteristics to match specialized transducers — and Foxboro engineers rely on *the* bridge that can measure all these characteristics accurately . . . the G-R Type 1603-A Z-Y Bridge.

If your requirements call for flexible impedance measuring equipment, consider the General Radio Z-Y Bridge . . . the Bridge that gives you  
*built-in versatility at no extra cost.*



## GENERAL RADIO Company

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**WE SELL DIRECT.** Our District Sales Offices are staffed by engineers especially trained to help you in the selection of instruments and measuring systems best suited to your needs. We welcome your inquiries — will help solve your problems.

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Eight additional Olsen UTMs (13 in all) are used throughout the vast Jenkintown, Pa. plant for constant quality control. Test results are obtained quickly—when and where needed. SPS personnel especially like the ease of operation and dependable accuracy that typify all Olsen testing equipment.

Whatever your requirements, you too can count on Tinius Olsen for utmost reliability in testing. Write for Bulletin 54.



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